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ENGINEERING CHEMISTRY:

A MANUAL OF

QUANTITATIVE CHEMICAL ANALYSIS.

FOR THE USE OF

STUDENTS, CHEMISTS AND ENGINEERS.

—BY—

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OF TECHNOLOGY.

WITH ONE HUNDRED AND FIFTY-FOUR ILLUSTRATIONS.

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PREFACE.

The preparation of this manual has resulted from many years of experience in the chemical laboratory, the work of which has been closely connected with engineering, and with the teaching of these subjects to students.

A treatise of this character cannot be too comprehensive in the treatment of a subject, nevertheless better results are obtained, from students, by arranging the matter in such a way that the principles and methods of work are indicated and then references given for further study and research.

Students commencing quantitative chemical analysis can with profit perform the first eleven exercises given in this work, with proper supervision, and then select a course of study suitable to their advancement: either for iron and steel chemistry; railroad laboratory practice; the technical application of water supply; the chemical technology of fuels, etc., etc.

It will be found conducive to thorough work, that each student before finishing any investigation, be required to write not only the analytical data, but also the references to the literature bearing upon the subject examined, following the plan outlined in the manual.

The articles upon gas analysis and valuation, blast furnace practice, the heating value of fuels, the purification of water for technical purposes, lubrication, car illumination, and the examination of Portland cement, have received especial attention, since these topics, at the present time, form a considerable portion of the work and investigations of engineers.

The following articles have been contributed by experts in each line of study:

"Blast Furnace Practice," by Edward A. Uehling, M.E.

"Determination of the Heat Balance in Boiler Tests," and contributions of portion of the article upon "Pyrometry," by Wm. Kent, M.E.

69624

"Carbon Compounds of Iron," by G. C. Henning, M.E.
"Practical Photometry," by Alten S. Miller, M.E.
"Electrical Units," by Albert F. Ganz, M.E.
"Energy Equivalents," by E. J. Willis, M.E.

The author has endeavored to acknowledge every excerpt made by him, with the proper reference thereto, and his thanks are due to those chemists from whose experiences valuable methods of analysis have been incorporated in the manual.

THOMAS B. STILLMAN.

STEVENS INSTITUTE OF TECHNOLOGY,
HOBOKEN, N. J., Dec. 31, 1896.

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List of Illustrations.

ERRATA.

Page 80, line 14, *for* "NO_2" *read* "NO_3."

Page 81, line 19, *add* "with dilute acetic acid."

Page 82, line 22, *for* "1000 parts of salt" *read* "1000 parts of water."

Page 119, line 16, *for* "hydroscopic" *read* "hygroscopic."

Page 125, line 10, *for* "hydroscopic" *read* "hygroscopic."

Page 170, line 22, *for* "Leduber" *read* "Ledeber."

Page 187, line 12, *for* "fidelity" *read* "fluidity."

Page 256, line 30, *for* "for carbon dioxide 24.2 per cent." *read* "carbon monoxide 24.2 per cent."

Page 259, line 21, *for* "$C_2H_4 = 11900$ calories" *read* "$C_2H_4 = 11911$ calories."

Page 263, line 3, *for* "(2.39 cubic foot of air)" *read* "(2.39 cubic feet of air)."

Page 270, line 21, *for* "827.62 B. T. U." *read* "754.6 B. T. U."

Page 271, line 21, *for* "1582. B. T. U." *read* "1391. B. T. U."

Page 273, line 36, *for* "1000.52 B. T. U." *read* "1115. B. T. U."

Page 288, line 25, *for* "pressed" *read* "passed."

Page 314, line 29, *for* "hydrogen oxide gas" *read* "hydrogen sulphide gas."

Page 373, line 3, *for* "24° Baumé at 60° F." *read* "24.7° Baumé at 60° F."

Page 433, line 19, *for* "Wilson's calorimeter" *read* "Wilson's colorimeter."

ENGINEERING CHEMISTRY.

QUANTITATIVE ANALYSIS.

I.

Determination of Iron in Iron Wire.

Weigh two samples of bright iron wire (each sample 0.500 gram); transfer to beakers (No. 3), add twenty-five cc. hydrochloric acid, five cc. nitric acid, cover the beakers with watch-glasses, and warm gently until solution is complete.

Proceed with each sample as follows: Add 100 cc. water, then ammonium hydroxide *gradually* until the solution is faintly alkaline; boil, filter upon a No. 4 ashless filter,[1] and wash precipitate with hot water until the washings no longer react alkaline. Dry at 105° C.

Remove as much of the dry precipitate as possible from the filter paper to a piece of glazed paper and ignite the filter paper in a weighed porcelain crucible (Meissen No. 6), uncovered, until all carbonaceous matter is consumed. Add the precipitate from the glazed paper, cover the crucible, and ignite at a red heat for ten minutes, cool in a desiccator, and weigh. Heat the crucible and contents once more to a red heat for three minutes, cool as before, and weigh. Repeat until weight is constant.

Example:

Amount of iron wire taken = 0.500 gram.

Crucible + Fe_2O_3	9.432 grams.
Crucible	8.721 "
Fe_2O_3	0.711 gram.

Then

$$Fe_2O_3 : Fe_2 :: 0.711 : x.$$

$$x = 0.4977 \text{ weight of Fe.}$$

$$\frac{0.4977 \times 100}{0.500} = 99.54 \text{ per cent. Fe in the wire.}$$

References.—Fresenius' "Quantitative Chemical Analysis" (London Edition), § 703, 1, *a.*; "Hints to Beginners in Iron Analysis," by David H. Browne, *J. Anal. Chem.*, 5, 325.

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[1] 12 cm. diameter.

II.

Alumina in Potash Alum.

$(K_2SO_4 + Al_2(SO_4)_3 + 24H_2O)$.

Press finely triturated potash alum between sheets of filter paper. Weigh out duplicate samples, each of two grams; transfer to No. 4 beakers, and dissolve in about 150 cc. of water.

Add ammonium hydroxide in slight excess, fifteen cc. solution of ammonium chloride, and boil gently a few minutes, the liquid remaining alkaline. Allow the precipitates to settle, then decant the clear supernatant liquid upon No. 4 ashless filters. Pour boiling water upon the precipitates in the beakers, allow precipitates to settle, decant the liquid as before, and repeat the operation three times, finally transferring all of the precipitates to the filter papers, and washing with hot water until the reaction is no longer alkaline. Dry at 105° C., transfer to weighed porcelain crucibles, and ignite as directed for ignition of ferric hydroxide (I).

Example:

Amount of alum taken, 2.384 grams.

Crucible + Al_2O_3	17.513 grams.
Crucible	17.258 "
Al_2O_3	0.255 gram.

$$\frac{0.255 \times 100}{2.384} = 10.69 \text{ per cent. } Al_2O_3.$$

Theoretical Percentage:

$$K_2SO_4 + Al_2(SO_4)_3 + 24H_2O : Al_2O_3 :: 100 : x.$$
$$x = 10.85 \text{ per cent. } Al_2O_3.$$

III.

Copper in Copper Sulphate.

$(CuSO_4 + 5H_2O)$.

About five grams of the crystallized salt are pulverized, pressed between folds of filter paper, and transferred to a small stoppered weighing tube, and the latter and contents accurately weighed.

Pour out about one gram of the salt into a No. 3 beaker, and reweigh the tube. The difference between the two weights gives the weight of the salt taken.

The salt is dissolved in about 100 cc. of hot water, and, if the solution is not clear, add a few drops of dilute sulphuric acid.

Warm gently, and add *gradually* a clear solution of sodium hydroxide, with constant stirring, until the reaction of the copper solution is alkaline; boil; the copper is precipitated as dark brown cupric oxide. Thus:

$$CuSO_4 + 2(NaOH) = \mathit{CuO} + Na_2SO_4 + H_2O.$$

The precipitate is allowed to settle, when, if sufficient sodium hydroxide has been added, the supernatant liquid will be colorless. Filter by decantation upon a No. 4 ashless filter, wash with hot water until reaction of washings is no longer alkaline, and dry at 105° C.

Remove the precipitate (as much as possible) from the filter-paper, and place it upon a piece of glazed paper.

The filter-paper (which will contain some cupric oxide) is transferred to a weighed porcelain crucible (No. 6 Meissen), and ignited.

A portion of the cupric oxide is reduced to copper by the incandescent carbon of the filter-paper. Allow to cool, add two or three drops of nitric acid, warm gently to dissolve the copper, and, when solution is complete, evaporate to dryness, and heat to redness, converting all the copper nitrate to cupric oxide. Add the rest of the cupric oxide remaining upon the glazed paper to the crucible, and heat, at red heat, to constant weight.

Example:

First weight of weighing tube and $CuSO_4 + 5H_2O$ ·	7.0250	grams.
Second weight of weighing tube and $CuSO_4 + 5H_2O$	5.9605	"
Copper sulphate taken	1.0645	"
Crucible + CuO	15.3744	"
Crucible	15.0360	"
	0.3384	gram.

$$CuO : Cu :: \text{wt. of } CuO : x \; (= \text{wt. } Cu)$$

$$79.5 : 63.5 :: 0.3384 : x$$

$$x = 0.2702.$$

Then,

$$\frac{0.2702 \times 100}{1.0645} = 25.38 \text{ per cent. of Cu.}$$

Theoretical Calculation :

$$CuSO_4 + 5H_2O : Cu :: 100 : x$$
$$249.5 : 63.5 :: 100 : x$$
$$x = 25.45 \text{ per cent. Cu.}$$

Found by Analysis : 25.38 per cent. Cu.

Difference : 0.07 per cent.

IV.

Volumetric Determination of Copper by Potassium Cyanide Solution.

Dissolve ten grams of potassium cyanide in 250 cc. of water and thoroughly mix.

Weigh out two grams of pure copper wire, transfer to a one-fourth liter flask, add twenty-five cc. nitric acid, warm gently until the copper is all dissolved ; boil to expel oxides of nitrogen; cool, dilute with water to the mark, mix well. Take fifty cc. of this copper solution, transfer to a No. 3 beaker, add ammonium hydroxide until the precipitate formed dissolves and the solution is alkaline.

Fill a fifty cc. burette with potassium cyanide solution, and gradually drop the cyanide solution into the copper solution until the blue color disappears and the solution becomes colorless.

Note the number of cc. of potassium cyanide solution required to do this, and mark upon the potassium cyanide bottle the value of one cc. in terms of copper. Thus :

Suppose fifty cc. of the copper solution required 31.3 cc. of potassium cyanide solution :

Then 31.3 cc. $KCN = 0.40$ gram Cu.
And 1 cc. $KCN = 0.0127$ gram Cu.

Having thus obtained the value of the potassium cyanide solution, it can be used for determining percentages of copper in alloys, bronzes, etc.

For example—Brass :

Two grams of brass are weighed out and treated with twenty-five cc. nitric acid, and the solution made up to 250 cc.

Fifty cc. of this solution is made alkaline with ammonium hydroxide, filtered, and the filtrate titrated with the potassium cyanide solution. Having determined the number of cc. of potassium cyanide solution required to decolorize the fifty cc. of the brass solution, the percentage of copper is calculated from above data.

Consult: Note on the use of potassium cyanide in the estimation of copper, by Geo. E. H. Ellis, F. C. S., *J. Soc. Chem. Industry*, **8**, 686.

V.

Determination of Copper by Electrolysis.

Weigh out five grams of crystallized copper sulphate, dissolve in 500 cc. water (preferably in a half liter flask), mix well.

Take fifty cc., transfer to a No. 2 beaker, and arrange the electrolytic apparatus as shown in Figure 1, connecting the weighed platinum cone N with the negative element of a Bunsen cell and the platinum spiral P with the positive.

Add a few drops of dilute sulphuric acid and water enough so that the solution in the beaker covers two-thirds of the platinum cone.

Copper is deposited upon the platinum cone and the deposition is generally complete in about four hours.

To determine when all the copper is precipitated, take out one drop of the *colorless* solution, in the beaker, by means of a glass rod, and place the drop upon a watch-glass. Bring in contact with this drop, one drop of a dilute solution of potassium ferrocyanide.

If copper is still unprecipitated, brown copper ferrocyanide will be formed. If, however, it is all precipitated, no brown coloration of the drops will form.

When the copper is all deposited remove the platinum cone quickly, wash it several times by dipping it in distilled water, dry at 100° C., and weigh.

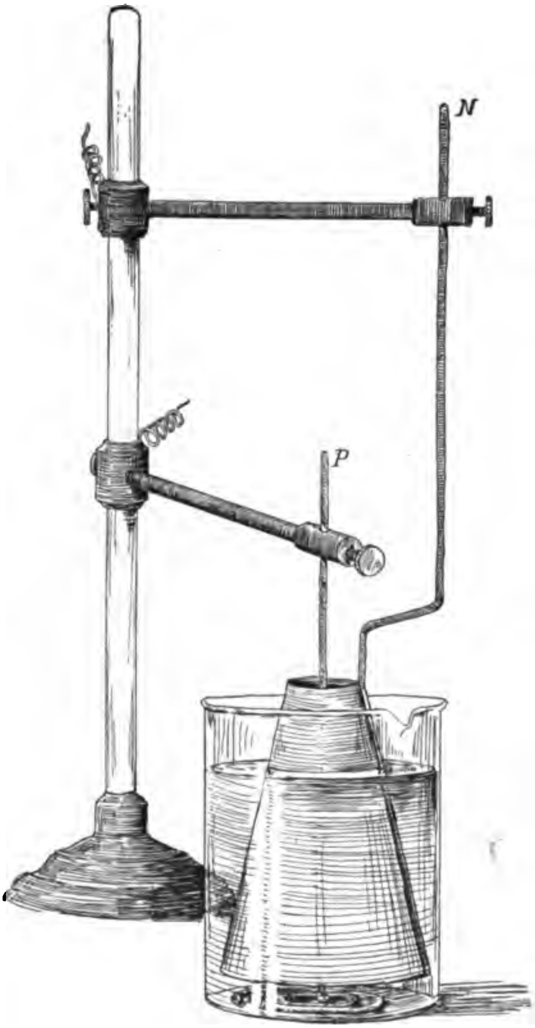

Fig. 1.

Example:

Amount of copper sulphate taken = 5.000 grams.
Solution 500 cc.
Fifty cc. taken for electrolysis.

Platinum cone + metallic copper............	36.656 grams.
Platinum cone	36.529 "
Copper deposited.........................	0.127 gram.

Then,

$$\frac{0.127 \times 10 \times 100}{5} = 25.40 \text{ per cent.}$$

Where many determinations of copper, by this method, are to be made, the apparatus described by W. Hale Herrick, *J. Anal. Chem.*, **2**, 67, can be used.

A very convenient instrument for generating the current of electricity is Gülcher's thermo-electric pile, Figure 2.

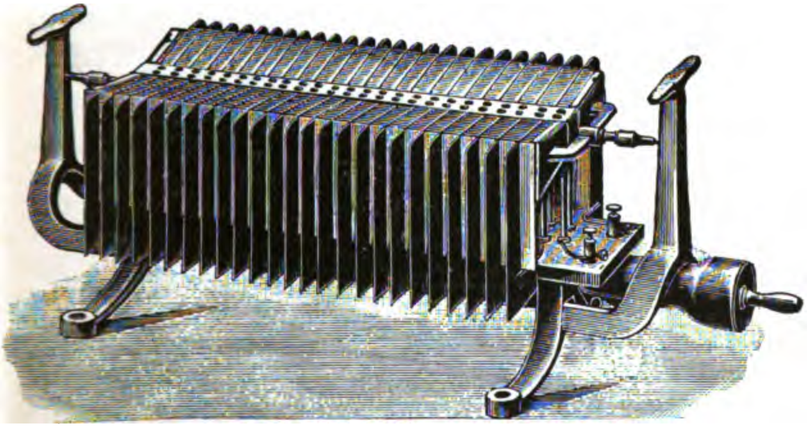

Fig. 2.

It consists of sixty-six elements and is equivalent to two large freshly filled Bunsen elements; its electromotive force is equivalent to four volts, the inner resistance amounting to 0.65 ohm, so that with an equal outer resistance the thermo-electric pile gives a current of three ampères. The gas consumption is about 170 liters per hour (6.001 cubic feet).

The amount of current should not be excessive, otherwise the deposit of copper upon the platinum cone will be granular and non-cohesive.

References: "Bibliography of the Electrolytic Assay of Copper," by Stuart Croasdale, *J. Anal. Chem.*, 5, 133-84.
"Electro-Chemical Analysis," E. F. Smith, p. 48.
"Quantitative Chemical Analysis by Electrolysis," by Dr. Alex. Classen, translated by W. Hale Herrick. 1894.
"The Utilization of the Electric Light Current for Quantitative Chemical Analysis," by P. T. Austen and J. S. Stillwell, *J. Anal. Chem.*, 6, 127.
"On the Analysis of American Refined Copper," by H. F. Keller, *J. Am. Chem. Soc.*, 16, 785.

VI.

Determination of Sulphur Trioxide in Crystallized Magnesium Sulphate.

Weigh out one and a half grams of crystallized magnesium sulphate. Transfer to a No. 3 beaker. Add 100 cc. water, a few drops of hydrochloric acid, and heat to boiling.

Add a solution of barium chloride in slight excess. Stir well, and set aside for half an hour.

Filter upon two No. 3[1] ashless filters, testing the filtrate with a few drops of barium chloride solution, to make certain that all the sulphur trioxide is precipitated.

$$MgSO_4 + BaCl_2 = BaSO_4 + MgCl_2.$$

Wash the precipitate thoroughly with hot water until a drop of the filtrate placed upon a watch-glass and brought in contact with a drop of solution of silver nitrate shows no turbidity. Dry the precipitate, and ignite in a weighed porcelain crucible to constant weight.

1st weight of tube + $MgSO_4 + 7H_2O$	7.9040 grams.
2nd " " " " "	6.5435 "
$MgSO_4 + 7H_2O$ taken	1.3605 "
Crucible + $BaSO_4$	23.502 grams.
Crucible	22.214 "
$BaSO_4$	1.288 "

$$BaSO_4 : SO_3 :: 1.288 : x$$

$$x = 0.442 \text{ gram } SO_3.$$

$$\frac{0.442 \times 100}{1.3605} = 32.48 \text{ per cent. } SO_3.$$

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[1] 9 cm. in diameter.

Theoretical:

$$MgSO_4 + 7H_2O : SO_3 :: 100 : x$$
$$x = 32.52 \text{ per cent. } SO_3.$$

References: Fresenius, "Quant. Chem. Analysis," §132, 1.
"The Volumetric Estimation of Sulphates," by D. Sidersky *J. Anal. Chem.*, 2, 417.,

VII.

Determination of Lead in Galena,

Transfer two grams of the finely powdered ore to a four-inch porcelain capsule; add twenty-five cc. nitric acid, warm, then fifteen cc. sulphuric acid, and evaporate *carefully* until red fumes cease to be evolved, and the residue is nearly dry.

Allow to cool, add a few drops of dilute sulphuric acid and seventy-five cc. water, bring to a boil, filter, and wash well. Neglect the filtrate. Wash the precipitate from the filter into a No. 3 beaker, using not over seventy-five cc. water; add 100 cc. of a solution of sodium carbonate in water, (1 to 10)and boil the contents of the beaker for fifteen or twenty minutes. Solution must be strongly alkaline.

By this action the lead sulphate, formed by the nitric and sulphuric acids upon the sulphide, is converted into carbonate. Filter, wash well with boiling water until reaction of washings is no longer alkaline. Neglect the filtrate.

Wash the precipitate into a No. 3 beaker with about seventy-five cc. of water, add seventy-five cc. strong acetic acid, warm, and keep the contents of the beaker at boiling temperature for ten minutes, beaker covered with a watch-glass.

The lead carbonate is thereby decomposed and soluble lead acetate formed, while any silica or gangue remains insoluble. Filter, wash well with hot water until the washings are no longer acid. Neglect the residue on the filter.

To the solution of lead in the beaker, which should not exceed 150 cc. or 200 cc., including the washings, dilute sulphuric acid is added in slight excess until no more precipitate is formed.

After standing for half an hour the lead sulphate is filtered off upon a No. 3 ashless filter, and washed thoroughly with hot water.

Dry at 102° C. Transfer the lead sulphate from the filter-paper to glazed paper, and ignite the filter-paper in a weighed porcelain crucible. After complete incineration, allow to cool; add a few drops of nitric acid, and warm *gently*. (Any lead reduced from lead sulphate by the burning paper will be dissolved, forming lead nitrate.) Add three or four drops of sulphuric acid and evaporate to dryness; add the rest of the lead sulphate that is upon the glazed paper, and ignite contents of the crucible to redness; cool in desiccator, and weigh; repeat to constant weight.

Example:

1st weighing of tube and Galena	16.670	grams.
2d " " " "	14.503	"
Galena taken	2.167	"
Crucible + $PbSO_4$	17.576	grams.
Crucible	16.564	"
	1.012	"

$$PbSO_4 : Pb :: 1.012 : x$$
$$x = 0.6914.$$

$$\frac{0.6914 \times 100}{2.167} = 31.9$$ per cent. lead in the sample of Galena.

VIII.

Determination of Iron by Titration with Solution of Potassium Bichromate.

a. *Where the Iron Solution is in the Ferrous Condition.*

Take one and a half grams of crystallized ammonium ferrous sulfate; transfer to a No. 3 beaker, and dissolve in 100 cc. of cold water; add ten cc. hydrochloric acid.

Make a solution of potassium bichromate by dissolving 14.761 grams of the "C. P." salt in 1,000 cc. water; mix well.

Each cc. is equivalent to 0.0168 gram of iron. (Consult Fresenius, "Quant. Analysis, London edition, § 112 *b*.)

Fill a fifty cc. burette with some of this solution, and drop the bichromate slowly into the beaker containing the iron solution until a drop of the latter placed upon a white porcelain slab and brought in contact with a drop of a very dilute solution of potassium ferricyanide no longer produces a blue or greenish coloration, showing the ferrous salt to be all oxidized to ferric salt. Note the number of cc. of the bichromate solution required to do this, and calculate percentage of iron in the ammonium ferrous sulphate.

Example:

Ammonium ferrous sulphate taken............. 1.503 gram.
12.27 cc. bichromate solution required to oxidize.
1 cc. = 0.0168 gram iron.
Then, 12.78 cc. = 0.2147 gram iron.

$$\frac{0.2147 \times 100}{1.503} = 14.28 \text{ per cent. iron.}$$

Theoretical percentage:

$$(NH_4)_2SO_4.FeSO_4 + 6H_2O : Fe :: 100 : x$$
$$x = 14.28 \text{ per cent.}$$

b. Where the Iron solution Exists in the Ferric State.

As the use of bichromate requires the iron to be in the ferrous condition so as to be oxidized by the bichromate, the ferric salt is reduced to ferrous as follows:

Take one and a half grams of ferric sulphate,[1] transfer to a 200 cc. flask, dissolve in fifty cc. water, add ten cc. hydrochloric acid, and a few pieces of "feathered" zinc. All the zinc must be dissolved and the solution colorless before it can be titrated with the bichromate. It is essential in this process, that all the ferric salt be reduced to ferrous, otherwise the number of cc. of the bichromate used would give too low a result for the percentages of iron.

To keep the iron solution in the flask from oxidizing while it is being reduced by the hydrogen from the reaction of zinc and hydrochloric acid, several methods are available:

1st. Method described by Fresenius, in which carbon dioxide is passed through the flask during reduction (see § 112).

2d. The stopper of the flask is arranged to allow escape of the

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[1] Use ammonium ferric sulphate instead of ferric sulphate.

hydrogen generated by the dissolving of the zinc by the hydrochloric acid, but prevents inlet of air.

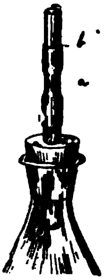

FIG. 3.

The stopper is of rubber (one perforation), through which passes a glass tube. At the upper end of the glass tube a piece of rubber tube (closed at *b* with a glass rod), is adjusted, and at *a* an opening is made in the rubber tube, which, when the contents of the flask are heated, allows the exit of gas, but which closes and prevents the entrance of air when heat is removed, the so-called Bunsen valve.

3d. The method of Jones is the most expeditious where a number of reductions are to be made.—*J. Anal. Chem.*, **3**, 124.

Example:

Ferric sulphate taken .... 1.520 gram.
18.01 cc. bichromate solution required to oxidize.

Then, $\frac{18.01 \times 0.0168 \times 100}{1.520} = 19.90$ per cent. iron in ferric sulphate.

Theoretical Percentage:

$Fe_2(SO_4)_3 + 9H_2O : Fe_2 :: 100 : x$
$x = 19.92$ per cent. iron in ferric sulphate.

IX.

Determination of Phosphoric Anhydride in Calcium Phosphate.

Weigh out one gram of finely pulverized calcium phosphate, transfer to a six-inch porcelain capsule, add twenty cc. nitric acid, ten cc. hydrochloric acid, and evaporate nearly to dryness. Allow to cool, add twenty-five cc. nitric acid, seventy-five cc. water, boil, and filter into a one-fourth liter flask. Wash with water until reaction is no longer acid, and make solution and washings up to the containing mark by the addition of more water.

The reading must be taken with contents of flask at a temperature of 15.5° C. to be accurate.

Mix well, and take duplicate samples, each of twenty-five cc., transfer to No. 3 beakers, and treat as follows:

Concentrate by evaporation to about fifteen cc. Cool somewhat, and add carefully ammonium hydroxide until the solution is alkaline, then make reaction slightly acid with nitric acid.

Add thirty cc. of standard ammonium molybdate solution, with stirring, and then some more ammonium hydroxide, but not enough of the latter to render the liquid alkaline. Add twenty cc. ammonium molybdate solution, and set aside two hours.

Filter, test filtrate with a few drops of ammonium molybdate solution, to be certain all of the phosphoric acid is precipitated, and wash precipitate well on the filter with water containing one-eighth its volume of ammonium molybdate solution.

The filtrate and washings are neglected.

Fifteen cc. ammonium hydroxide are poured upon the yellow precipitate on the filter, and the solution formed caught in a No. 2 beaker. The filter-paper, free from the yellow precipitate, is washed thoroughly with hot water, and the filtrate made acid with hydrochloric acid. This produces a precipitation of the yellow ammonium phosphomolybdate. Ammonium hydroxide is added in quantity just sufficient to dissolve this and to form a colorless solution again.

Thirty cc. of standard magnesia mixture solution are now added gradually with constant stirring, and the beaker with the precipitated ammonium magnesium phosphate set aside for thirty minutes.

Filter upon an ashless filter, wash with water containing one-eighth its volume of ammonium hydroxide, dry, ignite in porcelain crucible to constant weight, and weigh as magnesium pyrophosphate.

Example:

Amount of calcium phosphate taken = 1.157 grams.
Solution = 250 cc.

25 cc. taken.

Crucible + $Mg_2P_2O_7$	15.6037 grams.
Crucible	15.5210 “
$Mg_2P_2O_7$	0.0827 “

Then, $Mg_2P_2O_7 : P_2O_5 :: 0.0827 : x$

$x = 0.0529$ gram.

If the P_2O_5 in 25 cc. = 0.0529 gram, in 250 cc. or entire solution = 0.529 gram.

$$\therefore \frac{0.529 \times 100}{1.157} = 45.7 \text{ per cent. } P_2O_5 \text{ in } Ca_3(PO_4)_2.$$

References: A very complete article on "Mineral Phosphates and Superphosphates of Lime" will be found in the *American Chemist*, 7, 103–108; also

Bulletin, No. 89 (Oct. 9, 1892), "New Jersey Agricultural Experiment Station, Analysis and Valuations of Complete Fertilizers, Ground Bone, and Miscellaneous Samples."

J. Am. Chem. Soc., 15, 382.

J. Anal. Appl. Chem., 5, 418.

For method for complete Analysis of Phosphates and Superphosphates consult *Fres. Quant. Anal.*, p. 689. Also

Principles and Practice of Agricultural Analysis, H. W. Wiley, 2, 101–141.

X.

Determination of Chromium Trioxide in Potassium Bichromate.

Weigh out one gram of the finely crystallized salt, transfer to a No. 3 beaker; add 100 cc. of water, and warm until complete solution.

Take twenty-five cc. dilute hydrochloric acid, fifteen cc. alcohol, add to the solution of bichromate, and heat the mixture nearly to boiling, until the chromium trioxide is entirely reduced to chromium sesquioxide, the solution becoming dark green in color, then boil out the alcohol, and add ammonium hydroxide to faint alkaline reaction. The mixture is exposed to a temperature approaching boiling, until the liquid above the precipitate is perfectly colorless, presenting no longer the least shade of red.

Filter, wash with hot water until the washings no longer react alkaline.

Dry, ignite, and weigh as chromium sesquioxide.

Example:

1st weight tube and salt	10.942	grams.
2d " " " "	8.902	"
$K_2Cr_2O_7$ taken	2.040	"
Crucible and Cr_2O_3	43.270	"
Crucible	42.230	"
	1.040	

This weight of Cr_2O_3 must now be converted into CrO_3.

$$Cr_2O_3 : (CrO_3)_2 :: 1.044 : x$$

$$x = 1.3705 \text{ grams.}$$

$$\frac{1.3715 \times 100}{2.040} = 67.23 \text{ per cent. } CrO_3.$$

Theoretical:

$$K_2Cr_2O_7 : (CrO_3)_2 :: 100 : x$$

$$295 : 201 :: 100 : x$$

$$x = 68.13 \text{ per cent.}$$

References:

Fresenius, Quant. Anal., § 106, 1 *a*.

Volumetric Determination of Chromic Acid, *J. Anal. Chem.*, 5, 297.

XI.

Analysis of Limestone.

Carbonate of lime is the principal flux used by the iron smelter, and as usually quarried, is called limestone.

The composition of this varies greatly; the pure crystallized variety may be designated as marble, which usually contains about ninety-eight per cent. calcium carbonate, the remainder being silica and iron oxide.

Limestone, as distinct from marble, often contains organic matter (especially if very dark in color), alumina, ferrous or ferric oxide, ferrous sulphide, calcium sulphate, and magnesium carbonate, with the calcium carbonate.

A small proportion of iron oxide is of advantage in the smelting process, but an excessive amount of magnesium carbonate is objectionable, as it requires a higher heat for fusion than calcium carbonate, and more fuel is necessary in the blast furnace.

Analysis of Limestone.

Weigh out five grams of the finely-powdered material, transfer to a six-inch porcelain capsule, add fifty cc. hydrochloric acid, gradually, and evaporate to dryness on a water-bath. Add twenty-five cc. hydrochloric acid, 100 cc. of water, boil and filter upon two counterpoised filters into a one-fourth liter flask. Wash well, make solution up to the containing mark and thoroughly mix.

Residue. — Dry at 100° C. and weigh on the tared filter—weight obtained equals organic matter plus insoluble silicious matter.

Ignite in weighed porcelain crucible, subtract the weight obtained from the total weight on tared filter. The difference is the organic matter.

Wt. of residue on filter, 0.342 gram.

Cru. & insol. silicious material. 9.243
Crucible........................ 9.002
SiO_2........................ 0.241

0.342 gram, total insol. matter.
0.241 " insol. silicious matter.
0.101 " organic matter.

$\frac{0.241 \times 100}{5} = 4.8$ per cent. SiO_2, etc.

$\frac{1.101 \times 100}{5} = 2.02$ per cent. organic matter.

Organic matter. SiO_2, etc.

Solution.—Al_2O_3.Fe_2O_3, CaO, MgO.

Take two samples each of fifty cc. Transfer to No. 3 beakers, make alkaline with ammonium hydroxide, then acid with hydrochloric acid, and finally alkaline again with ammonium hydroxide.

Warm, filter, and wash well with hot water.

Residue.

Al_2O_3,Fe_2O_3.

Dry, ignite, and weigh as oxides of iron and aluminum. Multiply wts. obtained by 5, then $x \frac{100}{5} =$ per cent.

Cru. & Al_2O_3, Fe_2O_3........ 11.273
" 11.259
0.014

$\frac{0.014 \times 5 \times 100}{5} = 1.4$ per cent.

Al_2O_3 Fe_2O_3

Filtrate.—Add solution of ammonium oxalate in slight excess. Set aside three hours, filter, and wash well, with water containing one-eighth volume of NH_4OH.

Residue.

CaC_2O_4.

Dry, ignite to constant weight, and weigh as CaO.

Cru. & CaO = 11.5836
Crucible = 11.1620
CaO 0.4216

$\frac{0.4216 \times 5 \times 100}{5} = 42.16$ per cent. CaO.

CaO.

Filtrate.—If solution exceeds 200 cc. in amount, concentrate to about that volume. Add a few drops of ammonium hydroxide, then solution of sodium phosphate in excess, with constant stirring for two minutes. This precipitate is treated as directed for magnesium phosphate in the analysis of bone phosphate. See scheme IX.

Cru. & $Mg_2P_2O_7$.......... 11.4188
Crucible.................... 11.2160
$Mg_2P_2O_7$................ 0.2028

Calculate to MgO.

$Mg_2P_2O_7$:$(MgO)_2$::Wt.$Mg_2P_2O_7$: x

$x = 0.0731$

$\frac{0.0731 \times 5 \times 100}{5} = 7.31$ per cent MgO.

MgO.

SO_3. — Transfer 50 cc. of the solution to a No. 2 beaker, warm, add solution of $BaCl_2$ in slight excess. Set aside two hours.

Filter through two No. 2 ashless filters, wash well with hot water, dry, ignite, and weigh as $BaSO_4$, and calculate to SO_3.

Cru. & $BaSO_4$.. 12.078
Crucible........ 12.006
$BaSO_4$...... 0.072

$BaSO_4 : SO_3 :: 0.072 : x$

$x = 0.025$

$\frac{0.025 \times 5 \times 100}{5} = 2.50$ per cent. SO_3.

SO_3.

P_2O_5.—Take 50 cc. of the solution, concentrate by evaporation to about 30 cc. and treat as directed under determination of P_2O_5 in analysis of calcium phosphate.

Scheme IX.

P_2O_5.

CO_2. See page 18.

CO_2.

Determination of Carbon Dioxide.

The U tube B (Fig. 4) contains water acidified with sulphuric acid. No more of the mixture should be placed in the tube than just sufficient to cover the neck at a′.

The U tubes C and D contain granulated calcium chloride. As this chemical often contains free lime, it is always advisable before connecting these tubes with the apparatus to first pass carbon dioxide gas through them to saturate any free lime and then aspirate with air, to exhaust all free carbon dioxide.

The U tubes E and F contain soda lime granulated, medium size, and are weighed carefully before using the apparatus.

The U tube G contains calcium chloride to absorb any moisture that might enter F from the water in the aspirator H.

Three grams of the limestone are transferred to the flask A, and the flask connected with the apparatus shown in figure 4.

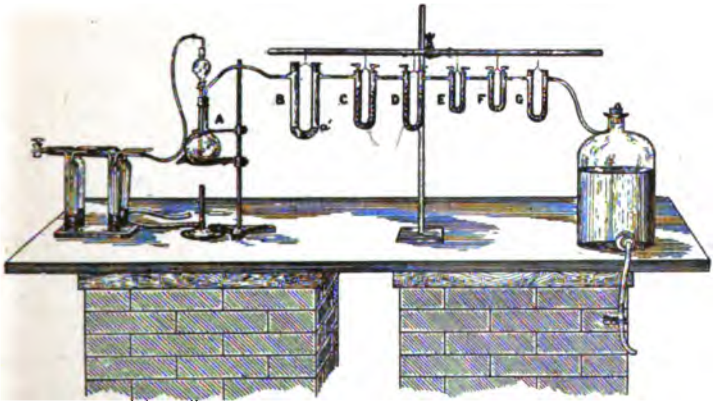

FIG. 4.

Dilute hydrochloric acid (fifty cc.) is allowed to run into the flask A from the funnel tube, and heat is gradually applied until the liquid in the flask begins to boil.

Connect the Bennert drying apparatus with the funnel tube of flask A and slowly aspirate air through the entire apparatus by means of the aspirator. The carbon dioxide is all absorbed by the soda-lime tubes.

After aspirating about four liters of air, weigh the soda-lime tubes to constant weight.

Soda-lime tubes and CO_2	48.2265	grams.
Soda-lime tubes	47.0320	"
CO_2	1.1945	"

$$\frac{1.1945 \times 100}{3} = 39.81 \text{ per cent. } CO_2.$$

Resumé:

Organic matter	2.02	per cent.
Silica	4.80	"
Iron and aluminum oxides	1.40	"
Lime	42.16	"
Magnesia	7.31	"
Sulphur trioxide	2.50	"
Carbon dioxide	39.81	"
	100.00	"

The SO_3 is united with CaO to form $CaSO_4$.

$$SO_3 : CaSO_4 :: 2.50 : x$$
$$x = 4.25.$$

Subtracting the 1.75 CaO used to unite with the SO_3 there remains 40.41 CaO to unite with CO_2.

$$CaO : CaCO_3 :: 40.41 : x$$
$$x = 72.71$$
$$MgO : MgCO_3 :: 7.36 : x$$
$$x = 15.36$$

Organic matter	2.02	per cent.
Silica, etc	4.80	"
Iron and aluminum oxides	1.40	"
Calcium sulphate	4.25	"
Calcium carbonate	72.17	"
Magnesium carbonate	15.36	"
	100.00	"

The analysis shows the limestone to be a dolomite or magnesium limestone. The following is an analysis[1] of high grade limestone:

Silica	0.87	per cent.
Iron and aluminum oxides	0.12	"
Calcium carbonate	98.60	"
Magnesium carbonate	0.22	"
	99.81	"

It is seldom that phosphoric acid is determined in limestone, since it usually amounts to less than two-tenths per cent. It is essential, however, in cases where the limestone is to be used in blast furnaces making Bessemer pig iron.

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[1] *J. Anal. Appl. Chem.*, 6, 510.

XII.

Coal and Coke Analysis.

Determination of Moisture, Volatile and Combustible Matter, Fixed Carbon, Ash, and Sulphur.

Take a weighed platinum crucible (capacity about twenty-five cc.) weigh in it one and a half grams of the powdered coal. Transfer to a drying oven and heat to 103° C. for fifteen minutes; cool in a desiccator, and weigh. Loss is moisture.

Crucible + cover + coal	26.117	grams
Crucible + cover	24.617	"
Coal taken	1.500	"

Water.

Crucible + cover + coal, before drying	26.117	"
Crucible + cover + coal, after drying	26.109	"
Moisture	0.008	"

$\frac{0.008 \times 100}{1.5} = 0.53$ per cent. moisture.

Volatile and Com. Matter.

The crucible containing the dried coal is now heated over a Bunsen burner for three and a half minutes, then over the blast-lamp for three and a half minutes more, taking care that the cover of the crucible fits closely. Cool in the desiccator. Loss in weight equals volatile and combustible matter plus one-half of the sulphur.

Crucible + cover + coal, before heating seven minutes	26.109	"
Crucible + cover + coal, after heating seven minutes	25.569	"
	0.540	"

$\frac{0.540 \times 100}{1.5} = 36.$ per cent.

Fixed Carbon.

The crucible and contents are now heated over a Bunsen burner (lid of crucible removed) until all carbonaceous matter is consumed. Where the combustion is extremely slow, it can be expedited by introducing into the crucible a slow current of oxygen gas so regulated that the contents of crucible are not disturbed. Replace cover of crucible when ignition is complete, cool in desiccator and weigh.

Crucible + cover + coal, before complete combustion	25.569	"
Crucible + cover + residue, after complete complete combustion	24.669	"
Fixed carbon + ½ S	0.900	"

$\frac{0.900 \times 100}{1.5} = 60.00$ per cent. Fixed carbon + ½ S.

Ash.

Crucible + cover + residue of coal after complete combustion (Ash)	24.669	grams.
Crucible and cover	24.617	"
Ash	0.052	"

$$\frac{0.052 \times 100}{1.5} = 3.46 \text{ per cent. ash.}$$

Resumé.

Moisture	0.53	per cent.
Volatile and combustible matter + $\frac{1}{2}$ S	36.00	"
Fixed carbon + $\frac{1}{2}$ S	60.00	"
Ash	3.46	"
Total	99.99	"

It is necessary, now, to determine the percentage of the sulphur present in the coal and subtract it from the amounts of volatile and combustible matter and fixed carbon.

The method is as follows:

Take one gram of the finely powdered coal, mix it, upon a piece of black glazed paper, with about ten grams of sodium carbonate (dry) and five grams of sodium nitrate.

Place a small portion in a platinum crucible of fifty cc. capacity, and heat to redness. When combustion is complete add some more of the coal mixture, repeating the operation until all has been transferred to the crucible from the glazed paper. Heat at a red heat for fifteen minutes, making certain that no particles of carbon remain unconsumed.

Allow to cool, transfer crucible and contents to a No. 3 beaker, add 100 cc. water, and warm carefully until the mass dissolves.

Remove the crucible from the beaker, washing it once with hot water, allowing the washings to run into the beaker. Filter the solution, acidify the filtrate with hydrochloric acid, boil, and add solution of barium chloride in slight excess. Allow to stand twelve hours, filter, wash well, dry, ignite, weigh as barium sulphate, and calculate to sulphur.

Thus:

Amount of coal taken	1.016	grams.
Crucible + $BaSO_4$	16.553	"
Crucible	16.511	"
$BaSO_4$	0.042	"

$$S = 0.0057 \text{ gram.}$$

$$\frac{0.0057 \times 100}{1.016} = 0.56 \text{ per cent. S.}$$

Taking this amount and subtracting one-half of it from the volatile and combustible matter of the coal, and one-half from the fixed carbon, the coal analysis will be :

Moisture	0.53	per cent.
Volatile and combustible matter	35.72	"
Fixed carbon	59.72	"
Sulphur	0.56	"
Ash	3.46	"
Total	99.99	"

In most cases the sulphur in coal exists combined with iron to form ferrous sulphide ; it also occurs as calcium sulphate, or both forms may be present in the same coal.

To determine the sulphur trioxide combined with the lime, take ten grams of the finely powdered coal and digest at a gentle heat, two hours, in a solution of sodium carbonate. It is filtered, washed with hot water, the filtrate made acid with hydrochloric acid, and the sulphur trioxide precipitated with barium chloride solution.

From the weight of barium sulphate obtained, the amount of sulphur trioxide is calculated.

Determination of Sulphur in Coal by the Eschka-Fresenius Method.

One gram of the finely powdered coal is mixed, in a platinum crucible, with twice its volume of a mixture of one part sodium carbonate and two parts of calcined magnesia, then heated in an uncovered platinum crucible until the mass becomes heated to a low red heat and the grey color of the mixture changes to a yellow or brownish-yellow hue. Allow to cool, treat with bromohydrochloric acid, filter, boil out the excess of bromine, and precipitate the sulphur trioxide with barium chloride solution, as barium sulphate and determine percentage of sulphur.

References :

Ann. Chem. (Liebig), 76, 90.
Ding. Polytech. J., 212, 403.
Am. Chemist, 6, 83.
J. Anal. Chem., 6, 86.
J. Anal. Chem., 6, 385.
J. Anal. Chem., 6, 611.
"On the manner in which Sulphur in Coal and Coke is Combined," by Dr. F. Muck, *J. Soc. Chem. Industry*, 6, 468.

Determination of Phosphorus in Coal and Coke.

Five grams of the powdered coal or coke are transferred to a platinum boat (Fig. 5). This boat is two inches square, one-half inch deep, and made from 0.002 platinum foil.

Care should be taken in making the boat that the corner flaps fit tightly, so that none of the ash will be lost by getting into the interstices.[1]

A tripod, Erdman chimney, and two pieces of platinum wire bent three-fourths of an inch below top of the chimney complete the apparatus.

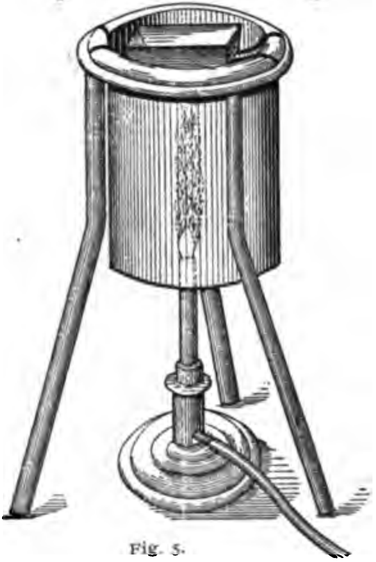

Fig. 5.

The heat applied for the first five minutes should be a low red, in order that none of the coal shall be lost in the escape of the volatile matter. After that the gas should be turned on full, and a bright red heat maintained. It is not necessary that the sample be ground very finely. After complete combustion of the carbon, the ash is transferred to a platinum crucible and fused with five grams of sodium carbonate and one gram of potassium nitrate. The fused mass is dissolved in forty cc. of dilute hydrochloric acid in a No. 4 beaker and evaporated therein nearly to dryness; thirty cc. of strong nitric acid are added and evaporated also nearly to dryness. The solution is then diluted with water, filtered from the silica, and the phosphoric acid precipitated with molybdate solution.

The analyses of a few representative coals are here given:

"BOG HEAD CANNEL" COAL.

Moisture	0.60	per cent.
Volatile and combustible matter	71.30	" "
Fixed carbon	11.20	" "
Sulphur	0.30	" "
Ash	6.60	" "
Total	100.00	" "

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[1] *Transactions Amer. Institute Mining Engineers*, 19, 66 [J. Lychenheim].

“Pittsburg Bituminous” Coal.

Moisture	1.28	per cent.
Volatile and combustible matter	37.36	“ “
Fixed carbon	57.33	“ “
Sulphur	0.72	“ “
Ash	3.31	“ “
Total	100.00	“ “

“Penn Anthracite,” Wilkes-Barre, Del. & Hudson Canal Co.’s “Vein No. 5.”

Moisture	4.182	per cent.
Volatile and combustible matter	4.283	“ “
Fixed carbon	85.320	“ “
Sulphur	0.794	“ “
Ash	5.521	“ “
Total	100.000	“ “

It is found in practice that coal from the same vein or seam varies in composition with the size of the coal; the percentage of ash increasing as the size of the coal diminishes. Thus, samples collected from the Hauto Screen building of Lehigh Coal and Nav. Co., Pa., gave the following:[1]

Size of coal.	Moisture.	Volatile matter.	Fixed carbon.	Sulphur.	Ash.	Total.
Egg	1.722	3.518	88.489	0.609	5.662	100
Stove	1.426	4.156	83.672	0.572	10.174	100
Chestnut	1.732	4.046	80.715	0.841	12.666	100
Pea	1.760	3.894	79.045	0.637	14.664	100
Buckwheat	1.690	4.058	76.918	0.714	16.620	100

These coals are separated into different sizes according to the mesh of the screen over which they pass. The sizes noted in the above table passed over and through sieve meshes of the following dimensions:

Broken or grate size	through	4.00 in.	over	2.50 in.
Egg “	“	2.50	“	1.75
Stove “	“	1.75	“	1.25
Chestnut “	“	1.25	“	0.75
Pea “	“	0.75	“	0.50
Buckwheat “	“	0.50	“	0.25

The composition of the ash of coal or coke is sometimes desired. The analysis can be made in a manner similar to scheme XIV.

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[1] *Transactions Amer. Inst. Mining Engineers*, 14, 720.

Analysis of a sample of ash of a Welsh coal, by J. A. Phillips. gave :

Silica	26.87	per cent.
Alumina and iron oxide	56.95	" "
Lime	5.30	" "
Magnesia	1.19	" "
Sulphuric acid	7.23	" "
Phosphoric acid	0.74	" "
Undetermined	1.72	" "
Total	100.00	" "

An analysis, by Gaultier, of the ash of a sample of English coke, gave the following :

Silica	42.10	per cent.
Alumina	34.40	" "
Calcium carbonate	4.80	" "
Magnesium carbonate	0.40	" "
Calcium sulphate	12.55	" "
Ferric oxide	5.28	" "
Total	99.53	" "

Coke is the best solid fuel for the blast furnace in the manufacture of pig-iron.

Charcoal, while having less ash, and producing combustion more readily, cannot be used in furnaces carrying large burdens, since it easily crushes and pulverizes.

Anthracite coal ignites and burns slowly in the furnace, and though it can withstand the burden, generally, without crushing, its slow work in the furnace has caused coke to supersede it.

The value of a coke is determined :

First, by chemical analysis ; a good coke showing a low percentage of ash, sulphur, and phosphorus, and a high percentage of fixed carbon.

Second, by mechanical tests, which comprise "Crushing Strength," "Porosity," specific gravity, etc.

The crushing strength can be determined by taking several samples of the coke, each one cm. high, and placing them in proper position in a Thörner compression machine, Fig. 6.

Good coke gives a compression strength of 160 to 175 kilos per cubic centimeter.

Connellsville coke usually gives a compression strength of 275. pounds per cubic inch.

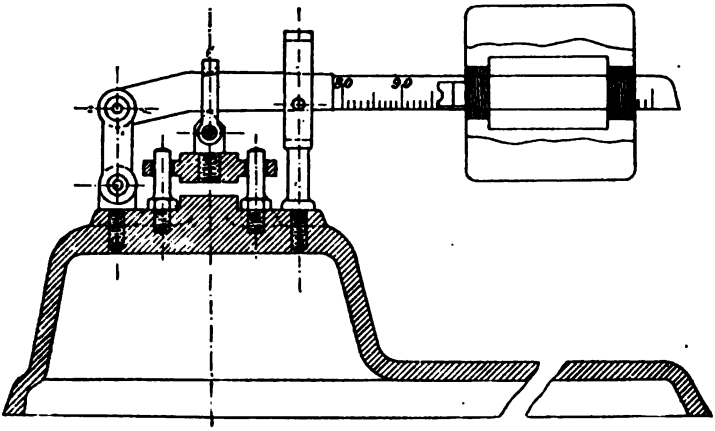

Fig. 6.

Porosity and specific gravity can be determined by the method used by Sterry Hunt in the Report of the Geological Survey of Canada, 1863, pp. 281–83.

This method is to select suitable specimens of any size or shape, generally between twenty and forty grams in weight, dry and weigh them, then fill their pores with water and weigh in water; the pieces are then taken out of the water, the excess of water upon their surfaces carefully removed, and weighed again in air. These three weighings furnish all the data necessary for calculating:

1. The apparent specific gravity, or the relationship between the whole mass of material and an equal volume of water.
2. The true specific gravity, or specific gravity of the particles.
3. The volume of pores in 100 volumes of material, or percentage of pores by volume.
4. The volume of pores in a given weight of material, as cc. in 100 grams.

The loss in weight of the material saturated with water when weighed in water, being equal to the volume of water displaced by the mass, enables us to determine the specific gravity of the latter; while this loss in weight, less the weight of the water absorbed by the mass, gives the true volume of water displaced by its particles, and hence the means of determining their specific gravity. The division of the amount of water absorbed by the amount of water displaced, gives the amount by volume of the pores in a unit of material, and the division of the weight of the water absorbed by the weight of the dry mass, gives the volume of pores in a unit of weight of the material:

Let $a =$ the weight of the dry material.
$b =$ the weight of the water which the material can absorb.
$c =$ the loss in weight in water, of the saturated material.

Then:

$c : a :: 1000 : a =$ the apparent specific gravity, or the specific gravity of the mass.
$c - b : a :: 1000 : a =$ true specific gravity, or specific gravity of the particles, water being 1000.
$c : b :: 100 : a =$ percentage by volume of the pores in the material.
$a : b :: 100 : a =$ volume of pores in 100 parts by weight of the material, say cc. in 100 grams.

In filling porous substances generally with water two methods are in use, one to soak the specimens in water for a time and then to place them in water under the receiver of an air-pump and exhaust until no more air is given off; and the other to keep them suspended in boiling water until the pores are filled with water, as is shown by their ceasing to gain weight on taking them out, cooling, and weighing.

A combination of both methods will be found advisable in experimenting with coke.[1]

A series of nine specimens from the Bradford Works of Frick & Co., yielded as follows:

	Moisture.	True sp. gr.	Apparent sp. gr.	Per cent. of cells by vol.	Cc. in 100 grams.
Maximum ……	0.096	1.79	1.033	54.37	66.31
Minimum ……	0.008	1.73	0.819	42.20	40.83
Average ……	0.034	1.76	0.802	49.37	55.73
Coke. El Moro, Colorado. Twelve samples.					
Maximum ……	0.225	1.85	1.047	54.66	71.36
Minimum ……	0.025	1.61	0.766	61.47	41.56
Average ……	0.114	1.69	0.919	45.75	50.39

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[1] *Fuels*, Mills and Rowan, pp. 149–150.

The following is a report upon a sample of Connelsville coke:

ANALYSIS OF THE COAL FROM WHICH THE COKE WAS MADE.

	Per cent.
Water	1.105
Volatile and combustible matter	29.885
Fixed carbon	57.754
Sulphur	1.113
Ash	9.895
	100.752

Analyst. A. S. McCreath.

ANALYSIS OF THE COKE.

	Per cent.
Water	0.030
Volatile and combustible matter	0.460
Fixed carbon	89.576
Sulphur	0.821
Ash	9.113
Total	100.000

Analyst. A. S. McCreath.

SPECIFIC GRAVITY, POROSITY, PER CENT OF CELLS, WEIGHT PER CUBIC FOOT, ETC. OF THE COKE.

Apparent specific gravity	0.892
True specific gravity	1.760
Per cent. of cells by volume	49.37
Volume of cells; cc. in 100 grams	55.73
Weight per cubic foot (lbs.)	55.68

COKE.

Method of Manufacture.					To be used for.	
Style of oven.	Size.	Charge in pounds.	Yield per cent.	Time of coking.	Kind of furnace.	Size of furnace.
Bee-hive	11 × 5′ 6″ 12′ × 6′	7600	63	48 and 72	Iron blast.	70′×16′

Tests made by J. H. Bramwell.

John Fulton, M. E., gives the following as the standard for the chemical and physical properties of coke:

FULTON'S TABLE EXHIBITING THE PHYSICAL AND CHEMICAL PROPERTIES OF COKE.

REVISED SERIES.

Locality.	Grams in one cubic inch.		Pounds in one cubic foot.		Percentage by volume.		Comp'sive strength per cubic inch. ¼ ultimate strength.	Height of furnace charge, supported without crushing.	Order in cellular space.	Hardness.	Specific gravity.
	Dry.	Wet.	Dry.	Wet.	Coke	Cells.					
Standard Coke. Connellsville.	15.47	23.67	58.98	87.34	49.96	50.04	301	120	1	2.5	1.89

Locality.	Chemical analysis.						Remarks.
	Fixed carbon.	Moisture.	Ash.	Sulphur.	Phosphorus.	Volatile matter.	
Standard Coke Connellsville.	87.46	0.49	11.32	0.69	0.029	0.011	

References: "On the Density of Coke," by Wm. A. Tilden, F. R. S., *J. Soc. Chem. Ind.*, 3, 610.

"An Investigation Regarding the Differences Between Cokes," by Sir I. Lowthian Bell, *J. Iron and Steel Institute*, 1885.

"The Physical and Chemical Properties of Coke," by John Fulton, *Transactions American Institute of Mining Engineers*,, 1885.

Grundlagen der Koks-Chemie, von Oscar Simmersbach, Berlin, 1895.

"A Method of Obtaining the Specific Gravity and Porosity of Coke," by W. Carrick Anderson, *J. Soc. Chem. Ind.*, 15, 20.

"An Investigation of Coals for Making Coke in Semet-Solvay Ovens," by J. D. Pennock, *J. Anal. Appl. Chem.*, 7, 135.

XIII. Commercial Analysis of Hematite, Limonite, Magnetite, and Spathic Iron Ores.

Take ten grams of the finely powdered ore, which has been dried at 105°C for one hour, transfer to six inch porcelain capsule, add fifty cc. hydrochloric acid, fifteen cc. nitric acid, and evaporate to dryness. Cool, add seventy-five cc. hydrochloric acid, warm, add one hundred cc. water, boil and filter into a one-half litre grad. flask. Wash well with water. Make contents of flask up to containing mark, the liquid being at a temperature of 15.5°C. Mix well.

Residue. (1)

Insol. mineral matter.

Dry, ignite and weigh as gangue.

If it is other than silica, fuse with Na_2CO_3 in platinum crucible. Cool, dissolve in water, acidify with HCl, evaporate to dryness in 3 inch capsule. Take up with $HCl+H_2O$, filter, wash well.

Residue.

SiO_2

Dry, ignite and weigh as such.

Solution.

Al_2O_3

Make alkaline with NH_4OH, warm, filter, wash well, dry, ignite and weigh as Al_2O_3

P_2O_5 (2)

Take two samples of the solution, each of 100 cc.: transfer to No. 3 beakers and evaporate to about 30 cc. Cool, add NH_4OH to alkaline reaction, then HNO_3 to faint acid, and pour in with constant stirring 40 cc. of standard ammonic molybdate solution. Allow to stand 3 hours, filter, test filtrate with more ammonic molybdate solution to make certain all the P_2O_5 is precipitated. Filter, wash with water containing ¼ its volume of the molybdate solution and treat the yellow precipitate as directed in scheme No. IX. Page 12.

Water of Hydration. (7)

Take 1.5 grams of the ore, dried at 105°C, transfer to a bulb tube and connect with Bennert drying apparatus. See Fig. 8. The "U" tube "B" contains $CaCl_2$. The tube and contents are carefully weighed and connected with the bulb tube "A" containing the iron ore. Heat is applied under the bulb "A" and gradually raised to red heat, air being slowly aspirated through the apparatus. The water of hydration of the ore is expelled and collected in "U" tube "B." After aspirating air through the apparatus 15 minutes slowly, weigh tube "B." Increase of weight=water of hydration.

Al_2O_3, Mn_3O_4, CaO, MgO. (3).

100 cc. of the solution are transferred to beaker (800 cc.) 500 cc. H_2O added, the liquid boiled. $(NH_4)_2CO_3$ (powdered) is slowly added until the solution becomes a dark red color, but the reaction must be faintly acid. Add about 25 cc. of a saturated solution $(NH_4)C_2H_3O_2$. Boil and filter.

Residue.

Dissolve in HCl, wash well and make solution up to 250 cc. Take 50 cc., add NH_4OH in slight excess, boil, filter, dry, ignite and weigh as $Fe_2O_3+Al_2O_3$. Then calculate the amount of Fe_2O_3 in this amount of solution from determination of Fe in (4). Subtract the weight of Fe_2O_3 from weight of $Fe_2O_3+Al_2O_3$, the difference is weight of Al_2O_3.

Solution.

Transfer to a flask, add a few drops of Br and of NH_4OH, shake until all the Br is dissolved, set aside 12 hours, filter, wash with water + 2 per cent. $(NH_4)C_2H_3O_2$.

Residue

MnO_2

Dry, ignite and weigh as Mn_3O_4.

Solution.

Add $(NH_4)_2C_2O_4$ and a few drops of NH_4OH.

Set aside 3 hours, filter and wash.

Residue

CaC_2O_4.

Dry, ignite and weigh as CaO.

Solution

Concentrate in a beaker to about 250 cc., add solution of Na_2HPO_4 in excess, with constant stirring, allow to stand 2 hours, filter, dry, ignite and weigh as $Mg_2P_2O_7$

FIG. 7.

Iron. (4)

Take two samples of the solution, each of 50 cc. transfer to flasks (150 cc.) having stoppers with Bunsen valves.

Reduce the ferric solution with zinc and titrate as directed in scheme VIII, page 11.

CO_2 (5)

3 grams of the ore are transferred to the CO_2 apparatus and the determination made as directed in analysis of Limestone. See scheme XI.

SO_3 (6)

Take 50 cc. of the solution, boil, add sol. $BaCl_2$ in slight excess, allow to stand 3 hours, filter through two ashless filters.

Wash with hot water, dry, ignite, and weigh as $BaSO_4$, and calculate to SO_3.

SiO_2	Al_2O_3 (Insol)	H_2O (hydrated).	Al_2O_3.	Mn_3O_4	CaO.	MgO.	Fe	$CO_2.SO_3$

Example:

Ten grams of iron ore taken.

Insoluble residue and crucible .... 10.551 grams.

Crucible .... 10.301 "

0.250 "

$\frac{0.25\times 100}{10}=2.50$ per cent. insoluble matter.

Solution = 500 cc.

Phosphorus pentoxide, (100 cc.)

a. Crucible + $Mg_2P_2O_7$ .... 8.923 grams.

Crucible .... 8.919 "

$Mg_2P_2O_7 = 0.004$ "

b. Crucible + $Mg_2P_2O_7$ .... 7.6140 grams.

Crucible .... 7.6105 "

$Mg_2P_2O_7 = 0.0035$ "

$Mg_2P_2O_7 : P_2O_5 :: 0.0038 : x$

$x = 0.0024$

$\frac{0.0024\times 5\times 100}{10} = 0.120$ per cent. P_2O_5.

$= 0.054$ " P.

Iron determination.

Fifty cc. reduced with zinc required 34.65 cc. standard $K_2Cr_2O_7$ solution. One cc. $K_2Cr_2O_7$ corresponds to 0.0168 gram iron. $34.65\times 0.0168 = 0.58212$ gram iron in fifty cc. of the iron solution.

Then $\frac{0.58212\times 10\times 100}{10} = 58.21$ per cent. Fe in the ore.

$= 83.16$ " Fe_2O_3 in the ore.

Sulphur Trioxide (50 cc.)

Crucible + $BaSO_4$ .... 11.126 grams.

Crucible .... 11.011 "

$BaSO_4 = 0.015$ "

$BaSO_4 : SO_3 :: 0.015 : x$

$x = 0.0051$

$\frac{0.0051\times 10\times 100}{10} = 0.51$ per cent. SO_3

Alumina (50 cc. from 250c.c. = 1-5 of 100 cc.)

Crucible + Al_2O_3,Fe_2O_3 .... 12.6614 grams.

Crucible .... 12.3160 "

$Al_2O_3,Fe_2O_3 = 0.3454$ "

Fifty cc. of the iron solution, by titration, gave 0.58212 gram of iron or 0.3326 gram of ferric oxide for fifty cc. of the 250 cc. solution of $Fe_2O_3Al_2O_3$ in (4) scheme XIII. Subtract this weight (0.3326) from weight of alumina and ferric oxide, (0.3454) in the fifty cc. The remainder equals 0.0128 grams alumina.

$$\frac{0.0128 \times 25 \times 100}{10} = 3.20 \text{ per cent } Al_2O_3.$$

Another method of determination of alumina in presence of ferric oxide, where the aluminum oxide is in small amount, is to fuse the weighed oxides with potassium hydroxide in a silver capsule, and extract with water. The alumina forms a soluble salt whereas the ferric oxide remains undissolved.

Filter off ferric oxide, wash, ignite and subtract weight from former weight of both oxides. Difference is weight of alumina.

Manganese oxide (100 cc.)

Crucible + Mn_3O_4	12.166 grams.
Crucible	12.131 "
Mn_3O_4 =	0.035 "

$$\frac{0.35 \times 5 \times 100}{10} = 1.75 \text{ per cent. } Mn_3O_4.$$

Lime (100 cc.)

Crucible + CaO	8.936 grams.
Crucible	8.929 "
	0.007 "

$$\frac{0.0027 \times 5 \times 100}{10} = 0.35 \text{ per cent. CaO.}$$

Magnesia (100 cc.)

Crucible + $Mg_2P_2O_7$	8.929 grams.
Crucible	8.919 "
$Mg_2P_2O_7$ =	0.010 "

$$Mg_2P_2O_7 : (MgO)_2 : : 0.010 : x$$

$$x = 0.0036$$

$$\frac{0.0036 \times 5 \times 100}{10} = 0.18 \text{ per cent. MgO.}$$

Water of Hydration.

Amount of ore taken	1.267 grams.
$CaCl_2$ tube + H_2O	29.065 "
$CaCl_2$ tube	28.963 "
H_2O=0.102	"

$$\frac{0.102 \times 100}{1.267} = 8.05 \text{ per cent. } H_2O \text{ (hydrated)}$$

Carbon dioxide absent.

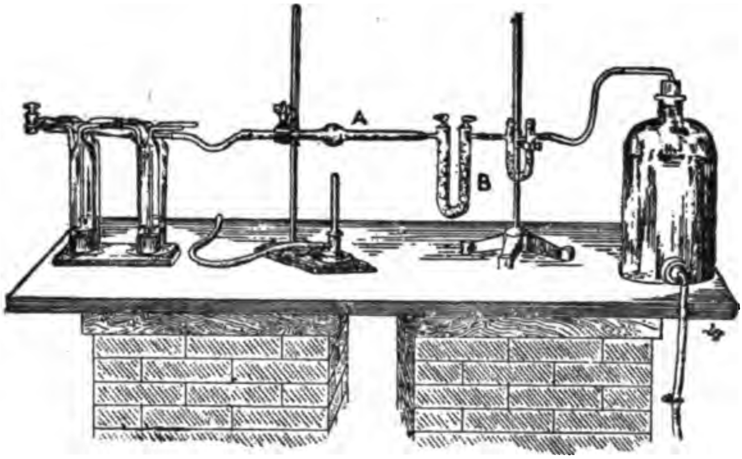

FIG. 8.

Resumé.

Insoluble mineral matter	2.50	per cent.
Al_2O_3	3.20	"
Fe_2O_3	83.16	"
Mn_3O_4	1.75	"
P_2O_5	0.12	"
SO_3	0.51	"
CaO	0.35	"
MgO	0.18	"
H_2O (hydrated)	8.05	"
Total,	99.82	"

If the ore is a magnetite, the iron exists as FeO, Fe_2O_3. There are several methods of determining the FeO in presence of Fe_2O_3. The one recommended by Whittlesay & Wilbur,[1] is frequently used, but the method of Allen is simpler and is to be preferred. It is as follows:

One gram of the very finely powdered iron ore is heated in a small sealed combustion tube, half full of fuming hydrochloric acid (25 cc. of the acid being sufficient). The heating is first performed in the water bath for two or three hours, then in a hot-air oven at 150° C for four hours more.

The ore is thus completely decomposed and after cooling the tube, it is broken under water in a beaker, and the ferrous oxide immediately determined by titration with standard solution of potassium bichromate. The amount of ferrous oxide

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[1] *Chemical News*, **19**, 270.

subtracted from the total oxides, determined in another sample of the ore, gives the amount of ferric oxide.

Some iron ores resist solution in acids, in which case the scheme is modified as follows:

Two grams of the finely pulverized ore are fused with fifteen grams of fusion mixture ($Na_2CO_3 + K_2CO_3$) in a large platinum crucible for one hour. After cooling the fused mass is treated with boiling water, the contents transferred to a four inch porcelain capsule, made acid with hydrochloric acid (carefully), and evaporated to dryness, twenty-five cc. hydrochloric acid, five cc. nitric acid are added, warmed until solution of iron is complete, then fifty cc. of water added, and the solution filtered from the silica, etc. The analysis can now be finished by scheme XIII.

Determination of Chromium in Chrome Iron Ore.[1]

Take a half gram of the very finely divided mineral and intimately mix it with twelve grams of a mixture containing equal parts of dry sodium carbonate and barium dioxide, transfer to a large platinum crucible and fuse over the Bunsen burner for one hour. At the end of this time a quiet fusion is obtained and the decomposition is completed. The crucible is then placed in a beaker, covered with water, and hydrochloric acid added, a little at a time, till the mass is completely disintegrated. The crucible is then removed, the solution made strongly alkaline with caustic potash, and ten cubic centimeters of a five per cent. solution of hydrogen dioxide added to oxidize the small amount of chromium sesqui-oxide that may be present. The solution is now boiled for twenty minutes to remove any excess of hydrogen dioxide, made acid with hydrochloric acid, and the amount of chromic acid determined by the aid of a standardized solution of ferrous chloride, one cubic centimeter of which corresponds to 0.015 gram Cr_2O_3.

The usual method for the determination of chromium in chrome iron ores, is that of Genth's[2] which consists in the fusion of the finely divided ore with potassium bisulphate.

In detail as follows:

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[1] Process of Donath modified by L. P. Kinnicutt and G. W. Patterson. *J. Anal. Chem.*, 3, 132.

[2] *Chem. News*, 6, 31.

A half gram of the pulverized ore is fused in a platinum crucible with ten grams of potassium bisulphate for one hour. This is allowed to cool when five grams of dry sodium carbonate and one gram of potassium nitrate are added and the mass subjected to fusion for one half hour. After cooling the crucible is transferred to a No. 4 beaker and the contents treated with water. Filter, wash well, and evaporate the filtrate to dryness in a porcelain capsule after acidulating with hydrochloric acid. Treat with hydrochloric acid, filter, wash with hot water, and reduce the chromium trioxide to chromium sesquioxide by the addition of ten cc. of alcohol and boiling (consult scheme X). Filter, dry and ignite the precipitate, which may contain some alumina, etc., with a small amount of sodium carbonate and potassium nitrate in a platinum crucible; cool, dissolve the fused mass in water and transfer to a platinum capsule and evaporate to a syrupy consistency. Add gradually crystals of potassium nitrate and continue this until effervescence ceases, add ammonia to alkaline reaction and filter. This precipitate contains the alumina, etc., that might have been present in the first precipitation of the chromium sesquioxide.

The chromium trioxide in the filtrate is reduced to the sesquioxide by the addition of excess of solution of sulphurous acid. Boil, make faintly alkaline with ammonia and continue boiling for several minutes. Filter, wash well, dry, ignite and weigh as Cr_2O_3.

The following analyses indicate the varying amounts of chromium sesquioxide in chrome iron ores:

Place.	FeO	MgO	Cr_2O_3	Al_2O_3	SiO_2			Analyst.
1. Chester Co., Pa.	35.14	......	51.66	9.72	2.09	=	99.32	Seybert.
2. Baltimore	36.00	......	39.51	13.00	10.06	=	99.11	"
3. " massive	18.97	9.96	44.91	13.85	0.82	=	98.15	Abich.
4. " cryst	20.13	7.45	60.04	11.85	......	=	99.45	"
					Mn.			
5. Siberia	24.00	......	53.00	11.00	10.00, 1.00	=	100	Langier.
6. Roraas, Nor	25.66	5.36	54.08	9.02	4.83	=	78.95	"
7. Bolton, Ga	35.68	15.03	45.90	3.20	......	=	99.81	Hunt.
9. Lake Memphramagog, U. S.	21.28	18.13	49.75	11.30	......	=	100.46	"
10. Beresof, Sib.	8.42	6.68	64.17	10.83	0.91	=	101.01	Moberg.
					Ca.			
11. Baltimore	30.04	......	63.37	1.95	2.21, 2.01	=	99.06	Rivot.
12. Voltena, Tuscany	33.93	......	42.13	10.84	4.75	=	100.65	Bechi.
					Ni.			
13. Texas, Pa.	38.66	......	63.38	......	...... 2.25	=	104.32	Garret.

Reference.—"New process for the oxidation of chromium ores and the manufacture of chromates," by J. Massignon, *J. Anal. Appl. Chem.*, 5, 465.

Determination of Titanium in Iron Ores.

The method of Bettel[1] is generally used.

Fuse about half a gram of the finely powdered ore with six grams of pure potassium bisulphate in a platinum crucible at a gentle heat, carefully increased to redness, and continued till the mass is in tranquil fusion. Remove from the source of heat, allow to cool, digest for some hours in 150 cc. of cold distilled water (not more than 300 cc. are to be used, as it generally causes a precipitation of some titanic acid); filter off from the silica, dilute to 1200 cc., add sulphurous acid until all the iron is reduced, then boil six hours, replacing the water as it evaporates.

The titanic acid is precipitated as a white powder, which is now filtered off, washed by decantation, a little sulphuric acid being added to the wash water to prevent it carrying away titanic acid in suspension. Dry, ignite, allow to cool, moisten with solution of ammonium carbonate, re-ignite and weigh. The titanic acid is invariably obtained as a white powder with a faint yellow tinge, if the process has been properly carried out.

The table on the next page gives the composition of the principle varieties of iron ores.

References.—(Iron Ores.) "The Iron Ores of the United States. *Proceedings of the Iron and Steel Institute*, special volume, 1890, pages 68-91.

"Hints for Beginners in Iron Analysis," by David H. Brown, *J. Anal. Appl. Chem.*, 5, 325.

"Determination of Iron by Stannous Chloride," by R. W. Mahon, *Amer. Chem. Jonrnal*, 15, 360.

"The Volumetric Determination of Titanic Acid and Iron in Ores," by H. L. Wells and W. L. Mitchell, *J. Am. Chem. Soc.*, 17, 878.

"The Constitution of Magnetic Oxide of Iron," by W. G. Brown, *J. Anal. Appl. Chem*, 7, 26.

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[1] Crookes' "Select Methods," p. 194.

Composition of Various Iron Ores.

	Lake Superior Red specular.	Limonite. Kentucky.	Magnetite. New Jersey.	Chrome iron ore. Maryland.	Ilmenite. Connecticut.	Clay iron ore. New York.	"Black Band" Scotland.	Spathic ore. Stahlberg.
FeO	....	....	26.52	20.13	22.39	45.86	2.72	47.96
Fe_2O_3	90.52	69.93	63.18	....	53.71	0.40	40.77	....
MnO_2	trace	....	0.12	....	0.25	0.96	....	9.50
Al_2O_3	1.39	3.12	3.28	11.85	....	5.86	....	....
CaO	0.70	1.53	0.38	....	....	1.37	0.90	....
MgO	0.42	1.62	....	7.45	0.50	1.85	0.72	3.12
SiO_2	5.89	13.45	6.68	....	....	10.88	10.12	....
CO_2	....	....	....	....	....	31.02	26.41	39.19
P_2O_5	0.26	0.25	0.05	....	....	0.21	....	....
SO_3	0.05	....	0.01	....	....	0.10	....	....
H_2O	1.90	10.21	....	....	....	....	....	....
TiO_2	....	....	....	....	23.72	....	....	....
Cr_2O_3	....	....	....	60.04	....	....	....	....
Organic Matter	....	....	....	....	....	0.90	17.38	....
Total	100.00	100.11	100.22	99.47	100.57	99.41	99.02	99.77

XIV. Blast Furnace Slag Analysis.

Fuse two grams of the finely powdered slag with ten grams of sodium carbonate (anhydrous) and one gram of sodium nitrate in a platinum crucible for fifteen minutes over a Bunsen blast lamp. Allow to cool, transfer crucible and contents to a No. 4 beaker, add 100 cc. water, warm, extract contents of crucible and remove latter. Acidify liquid in beaker with hydrochloric acid carefully, transfer contents to a six-inch porcelain capsule and evaporate to dryness. Add fifty cc. hydrochloric acid, 100 cc. water, boil and filter into a quarter liter graduated flask. Wash well with hot water, and cool to 15.5° C., add water to containing mark and thoroughly mix.

Residue. SiO_2

Dry, ignite and weigh as SiO_2.

Crucible + SiO_2 = 17.585 grams.
" — = 16.720 "

SiO_2 = 0.865

$\frac{0.865 \times 100}{2}$ = 43.25 per cent. SiO_2.

SiO_2.

P_2O_5.

Take one sample of 50 cc. from the solution and treat for phosphoric acid as directed in scheme IX.

Crucible + $Mg_2P_2O_7$ = 11.00935 grams.
" — = 11.00879 "

$Mg_2P_2O_7$ = 0.00056 "

$Mg_2P_2O_7$; P_2O_5 : : .00056 : x
x = .0003594

$\frac{.000359 \times 5 \times 100}{2}$ = 0.09 per cent. P_2O_5

P_2O_5.

FeO.

Take one sample of 50 cc. from the solution and reduce with zinc. Determine amount of iron by titration with $K_2Cr_2O_7$. See scheme VIII *b* and calculate to FeO.

50 cc. require 0.058 cc. $K_2Cr_2O_7$ solution.
1 cc. $K_2Cr_2O_7$ solution = 0.0168 gram Fe.
50 cc. solution of slag = 0.000974 " "
250 cc. " " " = 0.004870 " "
Fe : FeO : : 0.00487 : x
x = 0.0062

$\frac{0.0062 \times 100}{2}$ = 0.31 per cent. FeO.

FeO.

Al_2O_3.

Take 50 cc. and determine Al_2O_3 as in an iron ore. See scheme XIII.

Crucible + Al_2O_3 + Fe_2O_3 = 11.94415 grams
{ Subtract Fe_2O_3 found by titration } = 0.00139 "

Crucible + Al_2O_3 = 11.94276 "
Crucible = 11.8790 "

Al_2O_3 = 0.06376

$\frac{0.06376 \times 5 \wedge 100}{2}$ = 15.94 per cent. Al_2O_3.

Al_2O_3.

MnO_2, CaO, MgO.

Take 50 cc., add 100 cc. H_2O, make solution nearly alkaline with Na_2CO_3, add $NaC_2H_3O_2$, boil and filter off hydrated oxides of iron and aluminum. The filtrate is transferred to a 250 cc. flask, a few drops of bromine added, set aside twelve hours, then filtered and washed.

Residue.

MnO_2

Dry, ignite and weigh as Mn_3O_4 and calculate to MnO_2.

Crucible + Mn_3O_4 = 11.87936 grams.
" — = 11.87900 "

Mn_3O_4 = 0.00036 "

$\frac{0.00041 \times 5 \times 100}{2}$ = 0.10 per cent. Mn_3O_4.

MnO_2.

Filtrate.

Contains the CaO and MgO. Add ammonium oxalate, a few drops of ammonium hydrate and set aside twenty minutes and filter. Wash well with $H_2O + \frac{1}{4}NH_4OH$.

Residue.

CaC_2O_4

Dry, ignite and weigh as CaO.

Crucible + CaO = 12.02484 grams.
" — = 11.87900 "

CaO = 0.14584 "

$\frac{0.14584 \times 5 \times 100}{2}$ = 36.46 per cent. CaO.

CaO.

Filtrate.

Contains the MgO. Precipitate as per scheme XI with Na_2HPO_4 solution.

Crucible + $Mg_2P_2O_7$ = 11.90253 grams.
" — = 11.879 "

$Mg_2P_2O_7$ = 0.02353 "
$Mg_2P_2O_7$: $(MgO)_2$: : 0.02353 : x
x = 0.00843

$\frac{0.00843 \times 5 \times 100}{2}$ = 2.13 per cent. MgO.

MgO.

S.

Take 50 cc. and precipitate with $BaCl_2$ solution. See page 8 and calculate to S.

Crucible + $BaSO_4$ = 11.92356 grams.
" — = 11.879 "

$BaSO_4$ = 0.04456 "
S = 1.53 per cent.

S.

Resumé

Lime (CaO)	36.46	per cent.
Magnesia (MgO)	2.12	"
Silica (SiO_2)	43.25	"
Alumina (Al_2O_3)	15.94	"
Ferrous oxide (FeO)	0.31	"
Sulphur (S)	1.53	"
Manganese Oxide (MnO_2)	0.09	"
Phosphoric Acid (P_2O_5)	0.09	"
Undetermined	0.21	"
Total,	100.00	"

Form of Blank used for Reporting Blast Furnace Slag Analyses.

SLAG.

Date	......	......	......	......	......	......
Furnace	......	......	......	......	......	......
Ores used	......	......	......	......	......	......
....	......	......	......	......	......	......
....	......	......	......	......	......	......
No. of Iron	......	......	......	......	......	......
Lime (CaO)	......	......	......	......	......	......
Magnesia (MgO)	......	......	......	......	......	......
Silica (SiO_2)	......	......	......	......	......	......
Alumina (Al_2O_3)	......	......	......	......	......	......
Oxide of Iron (FeO)	......	......	......	......	......	......
Calcic Sulphide (CaS)	......	......	......	......	......	......
Manganese Oxide (MnO_2)	......	......	......	......	......	......
Phosphoric Acid (P_2O_5)	......	......	......	......	......	......
....	......	......	......	......	......	......
....	......	......	......	......	......	......
....	......	......	......	......	......	......
Chemist or Laboratory	......	......	......	......	......	.....

Examples of Blast Furnace Slags Analyses.

	No. 1.[1]		No. 2.[2]	
FeO	0.270	per cent.	0.436	per cent.
SiO_2	45.460	"	35.000	"
Al_2O_3	16.590	"	14.362	"
CaO	32.805	"	45.370	"
MgO	1.080	"	1.398	"
MnO_2	0.083	"	trace	"
Sulphur } Sulphide of	1.571	"	1.875	"
Calcium. } Calcium	1.963	"	1.500	"
Phosphoric Acid (P_2O_5)	0.008	"	0.059	"
Undetermined Loss	0.070	"	.....	"
	100.000	"	100.00	"

Some varieties of slag are soluble in hydrochloric acid, in which case the analysis can be made by scheme XIII. This applies also to open-hearth slags, refinery slag, tap-cinder, mill-cinder and converter slag.

Basic slags, from the Thomas-Bessemer Process, often contain as high as thirty per cent. of phosphoric acid and require a somewhat different process of analysis. Thus:

One gram of the finely pulverized slag is fused with excess of sodium carbonate in a platinum crucible. Extract with water, acidify solution with nitric acid and evaporate in porcelain capsule to dryness. Take up with hydrochloric, dilute to half a liter and precipitate the phosphoric acid by the Acetate process.[3]

The precipitate is filtered, dissolved in hydrochloric acid, excess of nitric acid added, and the solution concentrated until the hydrochloric acid and acetic acids are expelled. The nitric acid solution is diluted to half a liter and two portions are taken (each 250 cc.) and the phosphoric acid determined in these by the molybdate method; see scheme IX.

Blast furnaces capable of producing 300 tons of pig iron per day are becoming the rule rather than the exception, while an output of 400 tons in twenty-four hours is often reached. To show the amount of material required every twenty-four hours to keep such a furnace running, we will assume as follows:

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[1] Slag made during the run of Alice furnace, on mixture containing Enterprise ore.

[2] Slag made at the Sloss furnace in June, 1886, on No. 1 foundry iron. (Consult "Transactions of American Institute of Mining Engineers," Vol. XVI, p. 148).

[3] Fresenius Quant, p. 409, § 134.

Height of furnace, eighty feet; internal diameters at the hearth, bosh and stockline respectively fourteen, twenty and seventeen feet; cubical contents, about 22,000 cubic feet.[1] To produce one ton of iron, would require, approximately, 160,000 cubic feet of air, engine measurement, which would be at the rate of 33,333 cubic feet per minute $\left(\frac{160,000 \times 300}{24 \times 60} = 33,333.\right)$

To deliver this quantity of air a blowing power of not less than 2,000 horse-power should be available and 200 horse-power more is required to hoist the stock and pump the water needed for cooling, etc. The blast should be heated from 1200° to 1400° F, and for this purpose four regenerative stoves twenty feet in diameter and seventy feet in height are employed. These stoves contain about 48,000 cubic feet of fire-brick, and are kept at such a temperature as will heat the blast to the desired degree, by burning in them the waste gases of the furnace. If we assume the ore smelted to contain sixty per cent. of iron and twelve per cent. of silica, it will require one and six-tenths tons of ore and 0.4 tons of flux to make a ton of iron, assuming that two per cent. of the silica be reduced and alloyed with the pig iron. It will further require one ton of fuel[2] to make a ton of iron, which, containing ten per cent. of ash, will require an additional amount of 0.15 ton of flux. Thus, for one ton of iron is required $1.6 + 0.40 + 0.15 + 1. = 3.15$ tons of solid material and $\frac{160,000}{13.77 \times 2000} = 5.81$ tons of air. In one day therefore, there would be $300 \times 8.96 = 2688$ tons of material passing through such a furnace. Supposing the flux to be carbonate of lime, and to contain two per cent. of silica and one per cent. of alumina, the furnace would produce $(0.55 - 0.03 \times 0.55)\ 0.56 + 0.03 \times 0.55 + 1.6 \times 0.01 + 1 \times 0.01 = 0.57526$ ton of slag per ton of pig iron, or $0.57526 \times 300 = 172.5$ tons per day.

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[1] "The Modern Blast Furnace," E. A. Uehling, *Steven's Indicator*, 8, p. 17.

[2] Well equipped and well managed furnaces using "lake ores" are making a ton of iron (2,240 lbs.) with 1,800 lbs. of coke, and in some instances the fuel consumption has been as low as 1,600 lbs. of coke.

Summing up:

Material charged into the blast furnaces per day:

Ore	480	tons.
Coke	300	"
Flux	165	"
	945	tons.
Blast	1743	"
Total	2688	"

Tapped from bottom of furnace in molten state:

Pig Iron	300	tons.
Slag	172.5	"
Total molten product	472.5	"

Gaseous product passing out at top of furnace:

Total blast	1743.00	tons.
Oxygen from ore	144.00	"
Gasified Carbon, as CO and CO_2,	246.00	"
Carbon dioxide from flux	70.42	"
Volatile matter in ore and fuel	12.08	"
Total gaseous product	2215.50	tons.

Thus it is shown that of the material charged into a blast furnace somewhat less than sixty-five per cent. is gaseous, while over eighty per cent. passes off in the form of gas.

In addition to the 2215.5 tons of gas there must be added an equal weight of air, or nearly so, since considerable excess is required for combustion.

Thus the chimney of a 300-ton blast furnace, when in full operation, discharges into the atmosphere every twenty-four hours about 4,450 tons of gaseous material, which is at the rate of over three tons per minute. The heat energy developed is enormous. In twenty-four hours fully 7,500,000,000 heat units are generated, which, if utilized in a first-class steam plant, would develop over 13,000 horse power. The average amount of solid and molten material contained in a 300-ton furnace is probably not far from 900 tons. The temperature varies from 3000° F, in the hearth, to 300° at the stockline. If the heat varied regularly the average temperature would be 1650° F; but since the stock becomes denser as it gets lower in the furnace, and also since a red heat reaches quite high up in the furnace, 2,000° is probably nearer the average temperature of the whole. The specific heat of such a conglomerate is not definitely known, but it will be between two-tenths and three-tenths; assume it to be 0.25. Hence, is obtained, for the heat stored away in the incandescent furnace stock, 900 × 2000 × 2000 × 0.25 = 908,000,000 heat units.

The lining of the furnace will weigh 800,000 lbs; its average temperature will not be less than 800 degrees; the specific heat of fire-brick, at that temperature, is about 0.18; therefore the amount of heat stored away in the lining is 800,000 × 800 × 0.18 = 115,000,000 heat units.

The regenerative stoves contain something like 48,000 cubic feet of fire-brick, which, at 150 lbs. per cubic foot, would make 48,000 × 150 = 7,200,000 lbs. The average temperature of the brick-work in these stoves, when the temperature of blast is carried at 1400° may be taken at 1000° and the specific heat of the brick-work at that temperature, at 0.20. Upon this basis, the heat stored away in the regenerative stoves amounts to 7,200,000 × 1000 × 0.2 = 1,440,000,000 heat units. Thus, in a blast furnace of 300 tons daily capacity, there are the following quantities of materials consumed and heat units developed:

Charged into the furnace:

Solid material at the top	945 tons.
Gaseous material (blast) at tuyeres	1743 "
Total charged	2688 "

Discharged from furnace:

Molten material from hearth	472.5 tons.
Gaseous materials, dust and fume	2215.5 "
Total discharge	2688.0 "

Heat energy developed:

From fuel consumed in twenty-four hours,	7,500,000,000 heat units.
Stored in the incandescent material contained in furnace	908,000,000 heat units.
Stored in regenerative stoves	1,440,000,000 "
Total heat energy stored	2,348,000,000 "

Thus the stored energy is equal to $\frac{2,348,000,000}{2,000.} \times 778 =$ 913,272,000 foot-tons of mechanical energy.

The mechanical energy developed during twenty-four hours in the process of smelting is $7,500,000,000 \times 778 = 5,835,000,000,000$ foot pounds, or at the rate of $\frac{5,835,000,000,000}{24 \times 60 \times 2000 \times 5,280} = 383$ mile-tons per minute.

When working well, a blast furnace gives but little evidence of the immensity of the force it contains ; it is only when "running off" that one realizes, in a measure, what a monster it is. It is furthermore quite evident that the process must be continuous, twenty-four hours a day and three hundred and sixty-five days in a year, from the beginning to the end of the blast, which may last from six weeks to as many years.

When in good condition a furnace may be stopped for twenty-four or even forty-eight hours, without serious consequences, and when properly prepared may be "banked" for months and started up again.

The Charging of Blast Furnaces.

The process of smelting in a blast furnace is, of necessity, a continuous operation. The raw materials, ore, fuel and flux, are charged in at the top, keeping the furnace practically full, and the molten metal and slag are tapped out at the bottom at intervals as required. The time necessary for a charge to pass through the furnace varies from ten to forty hours according to the cubic contents of the furnace, the character of the ore, and the relative quantity of air driven through the furnace in a unit of time. Easily reducible ores require less time than those of a refractory nature. The average time in modern furnaces may be taken at twenty hours. In view of this fact, and of the further fact that the effects of bad fillings do not become positively manifest until the badly proportioned or irregularly distributed charges have entered the zone of fusion, and also that the correction for such irregularity can only become effective in the same zone, it becomes very evident that serious consequences might result from bad filling before the remedy could have had time to act.

The proper charging of a blast furnace is, therefore, of the utmost importance. This fact has long ago been acquired by practical experience, and the success of blast furnace management very largely depends on the proper proportioning and distribution of the fuel, ore and flux, in charging the furnace.

Since the successful running of a blast furnace depends more directly upon proper charging than upon any other one thing, it may be profitable to inquire how a furnace should be charged to obtain the best results. To do this we must study the chemical reactions as well as the physical changes which take place within a blast furnace.

The first requirement is heat, which must not only be sufficient in quanity and intensity, but it must also be properly distributed. *a.* The temperature must be a maximum at the tuyere-line and a minimum at the stock-line. The former temperature must be higher than the fusing point of the iron and slag, and the latter should be below the point at which carbon dioxide is reduced to carbon monoxide by the fuel. *b.* Each horizontal layer of the contents should have practically the same temperature throughout its whole area. *c.* The temperature of these horizontal layers should be fixed at fixed heights.

The second requirement is an abundant supply of an efficient reducing agent. Since all the sensible heat in a blast furnace is due to the combustion of carbon to carbon monoxide at the tuyeres, except that brought in by the blast, and carbon monoxide, as we shall presently show, being the most desirable reducing agent, it follows that if the first requirement is fulfilled the second must be also.

The third requirement is that the ore and flux shall be so proportioned and mixed that the impurities of the former will assimilate with the latter and with the ash of the fuel and form a fusible slag.

Of the solid material charged at the top, over fifty per cent. passes off in the form of gas—first, by the evaporation of the hygroscopic and combined water; second, by the volatilization of the hydrocarbons of the fuel and the carbon dioxide of the flux and air; third, by the reduction of the ore, the oxygen combining with carbon, forming carbon monoxide, or with carbon monoxide forming carbon dioxide; and lastly, by the oxidation of the carbon of the fuel, which unites with the oxygen of the blast, forming carbon monoxide at the tuyeres, part of which in its upward course reduces the ore, as already stated, forming carbon dioxide, the remainder passing off as carbon monoxide with the other gaseous products.

The principle object in view in operating a blast furnace is to secure the best possible conditions for reducing, carbonizing and melting the iron contained in the ores to be smelted, and to do this with the greatest regularity and the least expenditure of fuel for the quality of iron desired.

The chemical phenomena which take place in a blast furnace are manifold and complicated, and not altogether understood; but for our present purpose it is necessary only to consider the two principal reactions, *viz.*: "Reduction" and "Oxidation;" the latter always generating, and the former absorbing heat.

One pound of iron in being reduced from its ore (Fe_2O_3) absorbs 3,396 heat units.[1] Carbon in being oxidized to carbon monoxide generates 4,466 heat units, and when another atom of oxygen is added, forming carbon dioxide, 10,078 heat units more are developed.

The reduction of ore to the metallic state may and does take place in three different ways:[2] First, by oxidizing the carbon at the tuyeres to carbon monoxide by the oxygen of the entering blast. The carbon monoxide thus formed, taking another atom of oxygen from the ore, forming carbon dioxide, which passes off as such; second, the carbon, taking direct from the ore two atoms of oxygen, forming carbon dioxide as which it escapes; third, the carbon, taking directly from the ore only one atom of oxygen, passing off as carbon monoxide. Although it is true that the conditions in a blast furnace do not permit the full realization of the first two modes of reduction, it is none the less a fact that all three modes take place side by side in the process of smelting; and it now remains to be seen which one is the most economical, and what can be done in the way of charging a furnace to realize that one to the fullest degree.

One pound of iron (Fe) in the form of hematite ore (Fe_2O_3) holds in combination three-sevenths pound of oxygen. One

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[1] 3,396 represents the mean of Dulong's Andrews' and Favre and Silberman's experiments.

[2] Prof. Gruner *Etude sur les Hauts Fourneaux.*

pound of carbon to become oxidized to carbon monoxide requires one and one-third pounds of oxygen, forming two and one-third pounds of carbon monoxide, which is capable of combining with one and one-third pounds more of oxygen, resulting in three and two-thirds pounds of carbon dioxide.

The heat absorbed in reducing one pound of iron from its ore is not affected by the mode of reduction, being in each case 3,396 heat units. The heat generated by the oxidation of the carbon by the first mode is

$\frac{3}{7} \times \frac{3}{4} \times 4466 = 1435.5$ heat units generated at the tuyeres,
and $\frac{3}{7} \times \frac{3}{4} \times 10078 = 3239.5$ " " in process of reduction.

Total, 4675 " " " "
3396 " absorbed " "

leaving 1279 " surplus.

By the second mode of reduction we have

$\frac{3}{7} \times \frac{3}{8} \times 14544 = 2337$ heat units generated,
3396 " absorbed,

leaving 1059 " deficiency.

By the third mode of reduction we have

$\frac{3}{7} \times \frac{3}{4} \times 4466 = 1436$ heat units generated,
and, as before, 3396 " absorbed,

leaving 1960 " deficiency.

Thus we see that in the first case we have a surplus of 1279 heat units, in the second case a deficiency of 1059 heat units, and in the third case a deficiency reaching 1960 heat units, making a difference between the first and third cases, for the same consumption of carbon, of 3239 heat units in reducing one pound of iron.

Time and space will not permit at this time to point out and formulate, quantitatively, the heat requirements in addition to that absorbed in the process of reduction, nor is it necessary for our present purpose.

The total carbon requirement varies with the nature and amount of impurities contained in the ore, the temperature and hygroscopic state of the blast, the size, shape and construction of the blast furnaces, etc., etc.; for our purpose it will suffice to denote the heat required for smelting, in addition to that absorbed in the process of reduction, by the symbol X. Subtracting from this, our + or — surplus, we get for the additional heat required to complete the process of smelting :

First case, X — 1279 heat units.
Second " X + 1059 "
Third " X + 1960 "

Now, since all the heat required to satisfy X (excepting what is carried in by the heated blast) must be generated by burning carbon at the tuyeres, where a higher oxidation than carbon monoxide cannot result,[1] we have only 4466 heat units available per pound of carbon consumed by the blast.

The weight of carbon consumed in reducing one pound of iron, according to the first mode of reduction, is $\frac{3}{7} \times \frac{3}{4} = \frac{9}{28}$ pounds of carbon, resulting, as already shown, in a surplus of 1279 heat units, available to meet the requirement of X, which surplus, expressed in weight of carbon, is equal to $\frac{1279}{4466} = 0.284$ pounds burnt at the tuyeres.

By the second mode of reduction we have $\frac{3}{7} \times \frac{3}{8} = \frac{9}{56}$ pounds of carbon oxidized, resulting in a deficiency of 1059 heat units. We have, however, in this case consumed only one-half as much carbon as in case first; making this the same, we get $\frac{9}{56} \times 4466 = 718$ heat units additional, which reduces the deficiency to $1059 - 718 = 341$ heat units. Expressed as above, we have available for X, $-\frac{341}{4466} = -0.077$ pound of carbon.

Similarly we get by the third mode of reduction : $\frac{3}{7} \times \frac{3}{4} = \frac{9}{28}$ pounds carbon oxidized, resulting in a deficiency of 1960 heat units, which, expressed in pounds of carbon available for X, would be $-\frac{1960}{4466} = -0.443$ pound of carbon.

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[1] Whenever oxygen meets carbon in excess, and at a sufficiently high temperature, carbon monoxide results at once. The supposition that carbon dioxide is always formed first, and then again reduced to carbon monoxide, has not yet been demonstrated. It is much more probable that, when oxygen comes in contact with incandescent carbon, that carbon monoxide is the first product; if another atom of oxygen is then at hand (*i. e.*, if oxygen is in excess, and the temperature is not above that of dissociation), carbon dioxide is immediately formed; but even admitting that carbon dioxide is first formed, it can only be of momentary existence in the hearth of a blast furnace, and does not alter the effect.

To simplify the comparison, let us suppose, for the moment, that X is completely satisfied by the first mode of reduction, in which case we would have $\frac{X}{4466} - 0.284 = 0.00$ pound of carbon required to complete the process of smelting one pound of iron.

By the second mode of reduction we would have $\frac{X}{4466} - (-0.077) = 0.363$ pound of carbon required to complete the process of smelting, and, according to the third mode of reduction we would have $\frac{X}{4466} - (-0.443) = 0.727$ pound of carbon required to complete the process of smelting one pound of iron.

From these few calculations it becomes evident that it is a matter of vital importance which mode of reduction prevails in the furnace, and that the first mode should be sought to be realized to the fullest extent, and the third mode should be avoided entirely if possible.

The first mode of reduction implies that no direct reduction shall take place, *i. e.*, no oxygen shall be abstracted from the ferric oxide by carbon, which necessitates that all the carbon shall reach the zone of combustion and be there burned to carbon monoxide, which, on its upward course, reduces the ore (Fe_2O_3) and becomes itself oxidized to carbon dioxide.[1]

Since the direct combination of the carbon of the fuel with the oxygen of the ore can only take place by actual contact, it does not require much meditation to arrive at the conclusion that such contact should be avoided as far as possible. This can be done to a limited extent in charging the furnace.

Calculation of Blast Furnace Slag.

Assume that we have a mixture of ores, of which the average composition is:[2]

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[1] This mode of reduction, the desirability of which was first scientifically established by Prof. Gruner in his *Etude sur les Hauts Fourneaux*, cannot be fully realized in the present form of blast furnace, because contact between fuel and ore cannot be entirely avoided.

[2] Rossi: *J. Am. Chem. Soc.*, 12, 321.

IRON ORE.

SiO_2	20.00	per cent.
Al_2O_3	3.20	" "
CaO	3.10	" "
MgO	2.60	" "
Fe_2O_3	70.00	" "
Mn_3O_4	0.20	" "
P_2O_5	1.05	" "
SO_3	0.10	" "
	100.25	" "

Metallic Iron = 50. per cent.

LIMESTONE.

SiO_2	6.00	per cent.
Al_2O_3	1.15	" "
CaO	30.00	" "
MgO	19.00	" "
CO_2	44.20	" "
	100.35	" "

COAL.

Anthracite coal containing 6.28 per cent ash. Of which ash, the composition, in the *coal*, in per cent, is:

SiO_2	3.35	per cent.
Al_2O_3	2.73	" "
CaO	0.10	" "
MgO	0.10	" "
	6.28	" "

We have decided to obtain a slag of such a character that the fusibility will be about that of a *sesquibasic slag*, that is, if preferred, of one in which oxygen ratio is 4 : 3. Looking at the table[1] we see that, for such a type, one of lime saturates 0.714 of silica, or one of silica takes up 1.400 of lime. Assuming any proper amount of coal per ton of ore smelted and, in most cases, 0.75 ton is all that is required, we have all the data necessary for our calculations. Transform *all the analyses into lime:*

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[1]

Composition.	Acid.	Sesqui-acid.	Neutral.	Sesqui-basic.	Bibasic.	Tri-basic.	Quadribasic
SiO_2 per cent	68.19	61.65	51.72	41.66	34.88	26.30	21.13
CaO per cent	31.81	38.35	48.28	58.34	65.12	73.70	78.87
O of SiO_2 : O of bases	4 : 1	3 : 1	2 : 1	4 : 3	1 : 1	2 : 3	1 : 2
1CaO saturates *SiO_2*	2.143	1.6071	1.0713	0.714	0.538	0.357	0.268
1SiO_2 " *CaO*	0.466	0.622	0.932	1.400	1.858	2.829	3.732

ORE.

$SiO_2 =$	20.00	per cent.
$Al_2O_3 = 3.20 \times 1.631$	5.22	" "
$CaO = 3.10$	3.10	" "
$MgO = 2.60 \times 1.40$	3.64	" "
$Mn_3O_4 = 0.20$	0.15	" "
	$CaO = 12.11$	" "

The ore is equivalent per ton to

SiO_2	20.00	per cent.
CaO	12.11	"

LIMESTONE.

SiO_2	6.00	per cent.
$Al_2O_3 = 1.15 \times 1.631$	1.87	" "
$CaO = 30$	30.00	" "
$MgO = 19 \times 1.40$	26.60	" "
	$CaO = 58.47$	" "

The limestone is equivalent per ton to

SiO_2	6.00	per cent.
CaO	$CaO = 58.47$	" "

COAL.

SiO_2	3.35	per cent.
$Al_2O_3 = 2.73 \times 1.631$	4.45	" "
CaO	0.10	" "
MgO	0.14	" "
	$CaO = 4.69$	" "

The coal is equivalent per ton to

SiO_2	3.35	per cent.
CaO	4.69	" "

Hence, as we use only three-fourths of a ton of coal per ton of ore, the coal used is equivalent to three-fourths of the above analysis, or:

SiO_2	2.52	per cent.
CaO	3.52	" "
$SiO_2 = 20 + 2.52 = SiO_2$	22.52	" "

per ton of ore; the coal and ores are equivalent to $CaO = 12.11 + 3.52 = 15.63$. Since, to make the proper silicate, one of lime takes up 0.714 of silica, the 15.63 of lime in coal and ores will take up: $0.714 \times 15.63 = 11.16$ per cent. of silica, leaving of *free silica* in the ore and coal 22.52 — 11.16 = 11.36 silica to saturate with limestone. The six per cent. of *silica of the stone* will require, at the rate of 1.400 pounds lime per pound of silica, 6 × 1.40 = 8.40 lime, leaving of *free lime* or the equivalent in the limestone, 58.47 — 8.40 or 50.07 *free lime*. We have to saturate in coal and ores, 11.36 free silica. At the rate of saturation adopted, it will take: 11.36 × 1.40 lime = 15.91 lime; as 50.07 free lime in one ton of limestone requires only 15.91 of lime to saturate the silica in coal and ores, we need only per ton of ore and three-fourths ton coal,

$$\frac{15.91}{50.07} = 0.317 \textit{ ton} \text{ of limestone.}$$

The charges are thus: one ton of ore, 0.75 ton of coal, 0.317 ton of limestone and, as the ore contains *fifty per cent. of iron*, *we require* two tons of ore, one-and-a-half tons coal and 0.634 ton limestone per ton of pig made.

The composition of the slag is:

Silica in ore and coal per ton of ore and per ¾ ton of coal	22.526	per cent.
In stone 6 × 0.317 ton	1.902	“ “
Total SiO_2	24.428	“ “
Lime in ¾ ton coal and 1 ton ore (per ton ore)	15.63	“ “
In stone 0.318 ton × 58.47 per cent =	18.59	“ “
Total lime	34.22	“ “

equivalent to

SiO_2 =	24.428	or reducing to	SiO_2 = 41.66	per cent.
CaO =	34.220	percentage:	CaO = 58.34	“ “
	58.648		Total, 100.00	“ “

exactly a sesquibasic silicate.

Using the preceding charges of ores, stone and coal, we should have every reason to expect a slag of the above composition or of one very close to it.

We have adopted one and one-half tons coal per ton of pig. If greater accuracy were necessary the preceding calculations could be made over again with the new charges in coal; but, practically, it is absolutely useless, the ash of coal entering, as it may be seen, as a small percentage into the general composition. With inferior cokes or anthracite it becomes an important factor not to be neglected but too often ignored. Cokes with fifteen per cent. of ash are not uncommon in certain localities.

As an example of the close coincidence between slags actually run from known calculated charges and the slag determined, *a priori*, we quote the following slag run in a furnace sixty feet high, sixteen feet bosh, running on hot blast 850° to 900° F. Pressure of blast, seven and one-half pounds, American furnace, anthracite coal. The analyses of materials were as follows:

	Ores. Per cent.	Stone. Per cent.	Coal. Per cent.
SiO_2	23.31	9.90	3.00
Al_2O_3	4.51	3.88	2.30
CaO	1.61	28.00	0.10
MgO	3.41	16.00	0.08
Alkalies	2.67	.....	.....
Mn_3O_4	traces	.....	.....
P_2O_5	0.31	.....	.....
S	0.08	.....	.....

Making the calculations proportionally to the quantity of the different materials charged, we have:

CHARGES:

	Iron ore, 924 lbs.	Dolomite, 378 lbs.	Coal, 588 lbs.	Total.
Containing:				
	Lbs.	Lbs.	Lbs.	Lbs.
Silica	215.38	37.42	17.64	270.44
Alumina	41.67	14.66	13.52	69.85
Lime	14.90	105.84	0.59	121.33
Magnesia	31.50	60.48	0.47	92.45
Alkalies	24.67	....	....	24.67
Mang. oxide	Traces	....	....	....
Amount of slag ingredients				578.74

Nine hundred and twenty-four pounds of ore gave in iron 425 pounds, the ores having 46.60 per cent. iron. With such slag, of which the character was sesquibasic, a light grade of iron was to be expected, such pig as contains in an average 1.50 per cent. silicon or 3.20 per cent. silica corresponding, in 425 pounds of pig iron, to 13.60 pounds of silica, which, subtracted from the total silica which went to form slag and pig, leaves a balance of 256.84 pounds silica to be expected in slag. The composition of the slag was then :

	Calculated. Lbs.	Per cent.
SiO_2	256.84	45.44
Al_2O_3	69.85	12.36
CaO	121.33	21.40
MgO	92.45	16.36
Alkalies	24.67	4.40
	565.14	99.96

The analysis gave:

	Per cent.
SiO_2	44.27
Al_2O_3	12.91
CaO	20.00
MgO	16.50
Alkalies	3.98
FeO	2.47
MnO	Traces
S	0.56
	100.69

This quantity of iron oxide, 2.47 per cent., is not abnormal, but occurs in many slags. If we take it into consideration in calculating the slag we have 99.96 + 2.40 = 102.36. Reducing to a percentage we find :

	Calculated Slag (iron added). Per cent.	Actual Analysis. Per cent.
SiO_2	44.34	44.27.
Al_2O_3	12.06	12.91.
CaO	20.88	19.81.
MgO	16.00	16.50.
Alkalies	4.22	3.98.
FeO	2.47	2.47.

The iron was found to be No. 3 light gray, containing 1.53 per cent. silicon. Transformed into lime, this slag corresponds to :

	Per cent.
SiO_2	40.66
CaO	59.34
	100.00

Types of Slags.

Chem. Denomination: *Acid.* Disilicates.	Chem. Denomination: *Sesquiacid.* Trisilicates.	Chem. Denomination: *Neutral.* Metasilicic Monosilicates.	Chem. Denomination: *Sesquibasic.* Disilicates.	Chem. Denomination: *Bibasic.* Orthosilicic Monosilicates.	Chem. Denomination: *Tribasic.* Parasilicic Monosilicates.
Oxygen Ratio: O of Silica : O of bases =4 : 1	Oxygen Ratio: O of Silica : O of bases =3 : 1	Oxygen Ratio: O of Silica ; O of bases =2 : 1	Oxygen Ratio: O of Silica ; O of bases =4 : 3	Oxygen Ratio: O of Silica: O of bases =1 : 1	Oxygen Ratio: O of Silica: O of bases =2 : 3.
Composition: Silica ....68.19 Lime 31.81	Composition: Silica ....61.65 Lime 38.35	Composition: Silica ....51.72 Lime 48.28	Composition: Silica ....41.66 Lime 58.34	Composition: Silica ....34.88 Lime............65.12	Composition: Silica ....26.30 Lime 73.70
Saturation: 1 Lime satur..2.143 SiO_2 1 Silica saturates.... 0.466 Lime	Saturation: 1 Lime .....1.607 Silica 1 Silica .....0.622 Lime	Saturation: 1 Lime .....1.071 Silica 1 Silica.....0.932 Lime	Saturation: 1 Lime .....0.714 Silica 1 Silica .....1.400 Lime	Saturation: 1 Lime .....0.538 Silica 1 Silica .....1.858 Lime	Saturation: 1 Lime .....0.357 Silica 1 Silica ....2.829 Lime
Fusibility: "Fusible."	Fusibility: "Moderately Fusible"	Fusibility: "Very Fusible."	Fusibility: "Very fusible," but less than preceding one.	Fusibility: "Fusible."	Fusibility: "Fusible," but less than preceding one.
Grade of iron likely to accompany such slag: Gray No. 2.	Grade of iron likely to accompany such slag: Gray, lighter.	Grade of iron likely to accompany such slag: White.	Grade of iron likely to accompany such slag: Mottled and Lighter Grays:	Grade of iron likely to accompany such slag: Gray No. 2, No. 2x, No. 2xx, and No. 1 occasionally.	Grade of iron likely to accompany such slag: Gray No. 1. Kish Cinder.

Graphic Method for Calculating Blast Furnace Charges.

The rule consists of two equal scales at right angles, Fig. 9, one of which (*a*) is fixed to a small board, while the other (*b*) is fixed at right angles to *a* upon a block, *c*, which is capable of sliding motion in a groove parallel to *a*.[1]

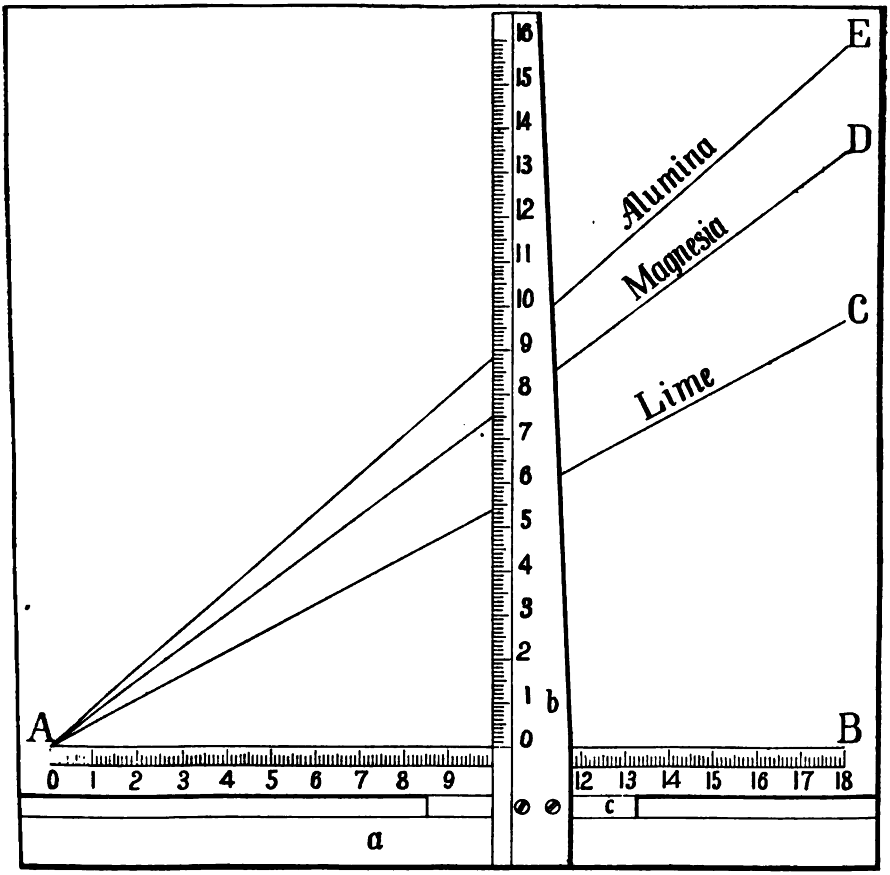

Fig. 9.

The point A, given by the intersection of the zeros of the scales, is marked upon the board, and from it a line AB parallel to the groove is drawn. With A as a centre, lines AC, AD, AE, are also drawn, making with AB, angles whose tangents are

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[1] H. C. Jenkins, *Iron and Steel Institute*, 1891.

equal to the ratios between the weight of the silica to weight of base in the respective silicates which it is desirable to produce in order to form the typical fusible slags ordinarily met with in blast furnace practice.

The lines AC, AD, AE are marked with the names of the bases for which they have been calculated.

Thus AC makes an angle of 28° 10′ with AB—this angle having a tangent whose value is 0.5357, which is the ratio of the atomic weight of silica to twice the atomic weight of lime, and corresponds to calcium silicate : this line, therefore, is marked "Lime."

Similarly the line AD makes an angle of 36° 52′ with AB, the value of whose tangent is 0.75, or the ratio of the atomic weight of silica to the atomic weight of 2 MgO : hence it is marked "Magnesia."

Also the line AE is at an angle of 41° 25′, and this having a tangent corresponding to the ratio of the atomic weight of 3 SiO_2, to that of 2Al_2O_3, makes the line correspond to the value of the component parts of silica and of alumina in aluminum silicate, and so it is marked "Alumina."

With such a scale it is a very simple matter to at once read off either the excess of silica in any ore, or else the amount required to properly flux off the earthy bases present.

As an example, take a spathic ore containing :

		Silica Required.	
FeO	50 per cent	....	per cent.
MgO	3 "	2.25	"
CaO	5 "	2.68	"
Al_2O_3	3 "	2.65	"
SiO_2	3 "	....	"
CO_2	36 "	....	"

Then setting the movable scale *b* against 3 on the fixed scale *a* and looking along *b* until the line marked "Magnesia" cuts it, we find the value 2.25 as being the amount of silica required to satisfy the magnesia. In like manner is found the amount (2.68) of silica required for the lime, and the amount (2.65) for the alumina respectively : adding all these together we find a total of 7.58 parts of silica required for every hundred of the ore.

But as there are already three parts present, every hundred parts of the ore require 7.58—3. = 4.58 parts of silica added to flux it.

Due allowance is also made for the ash of the coke, and any small quantity of sulphur in the mixture. In the treatment of several kinds of ores to be smelted together they should be mixed and divided into three classes, one having *less* and another *more* iron than is required in the final charge, and one should be *acid* and another *basic* after the correction for the ash of the coke is made, or one of these three may be a limestone or a siliceous flux: it need not necessarily contain iron.

Then let it be required to have n parts of iron per hundred of the charge, and let a_1, a_2, a_3 be the percentages of iron in the ores, and b_1, b_2, b_3 percentages of deficiency (or excess) of silica in the same, and x, y, z, the number of parts required of the component ores per hundred of the charge

FeO	SiO_2
x	$(a_1 + b_1)$
y	$(a_2 + b_2)$
z	$(a_3 \pm b_3)$

then,

$$(1)\quad x + y + z = 100.$$

$$(2)\quad \frac{xa_1 + ya_2 + za_3}{100} = n$$

$$(3)\quad xb_1 - yb_2 \pm zb_3 = 0$$

By solving these simple equations there is obtained, at once, the number of parts of each component required to satisfy the conditions of the charge.

If it is desired to produce a more acid or a more basic slag, it only requires that the scale b be replaced by one having a length of one-half (for bi-silicate slag), or twice (for bi-basic slag) that of the normal scale.

References:

"Note on the Determination of Silica in Blast Furnace Slag," by P. W. Shimer. *J. Am. Chem. Soc.*, 16, 501.

"Estimation of Metallic Iron in Slag" by G. Neumann, *Ztschr. anal. Chem.*, 6, 680.

"Estimation of Phosphoric Acid in Basic Slags" by V. Oliveri *J. Anal. Chem.* 5, 415.

"The Determination of Phosphoric Acid in Basic Slags" by Adolph F. Jolles, *J. Anal. Chem.*, 6, 625.

XV.

The Analysis of Water to Determine Scale-Forming Ingredients.

The scale-forming ingredients of a water are usually composed of calcium and magnesium carbonates and calcium sulphate, and though an analysis of a water for boiler purposes usually states the number of grains per gallon of the above constituents, the analysis should also comprise the determination of other ingredients, not scale forming, that are necessary to a proper estimation of the former. This is especially true of the alkalies, which are not always determined in a commercial analysis, of water, for boiler purposes; the amounts of lime, magnesia, chlorine, carbon dioxide and sulphuric acids, being considered a sufficient index of the character of the water.

The alkalies and their salts rarely form scale in boilers[1] and so cannot be classed as scale-forming, yet they play fully as important a part in the relation they sustain to the sulphuric acid and chlorine.

If all the sulphuric acid in a water were combined with the alkalies, there would be no sulphate of lime present, and the latter would be eliminated as a part of the scale ingredients. This is a condition rarely occurring, however, since in most waters a portion of the sulphuric acid is united with the alkaline earths and the alkalies. The indirect estimation of the carbon dioxide would be changed also. That is to say, where the carbon dioxide is estimated by uniting all the lime and magnesia (left uncombined with sulphur trioxide and chlorine), with carbon dioxide, it is evident that if all the sulphur trioxide is united

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[1] A sample of Boiler scale, from Charlestown S. C., analyzed by the author in 1887 had the following composition:

Carbon	1.01	per cent.
SiO_2	1.52	" "
Al_2O_3	0.43	" "
NaCl	72.12	" "
$CaCl_2$	10.32	" "
KCl	1.01	" "
$MgCl_2$	1.71	" "
$CaSO_4$	11.20	" "
Undetermined	0.68	" "
	100.00	" "

with lime, when a large portion belonged to the alkalies, the amount of calcium carbonate would be too small, and also that the proportion of the carbon dioxide would be deficient by the amount required to saturate the lime incorrectly united with the sulphur trioxide. There is nothing in the usual commercial analysis to indicate whether the sulphuric acid, as determined in the water, is all united with the lime to form calcium sulphate or not: but the custom has been so to unite it, with the result that calcium sulphate may be represented as a large component of the scale-forming material, when, in reality, none whatever may be present.

In a report of a partial analysis of the Monongahela River water, (*Transactions Amer. Inst. Mining Engineers*, Vol. XVII, p. 353), the amount of objectionable substances, for boiler use, are given as follows:

Total lime..... 161 parts per 100,000 = 94 grains per gallon.
" magnesia 33 " " " = 19 " " "
Sulphuric acid. 210 " " " = 122 " " "
Chlorine....... 38 " " " = 22 " " "

It further states the amounts of carbonates of lime and magnesia precipitated upon boiling to be:

Carbonate of lime...... 130 parts per 100,000 = 76. grains per gallon.
" " magnesia. 21 " " " = 12.2 " " "

The alkalies not having been determined, the proportion of sulphuric acid combined with them becomes problematical: in fact, the inference is that there are not any present, when in all probability, they may amount to a large percentage.

For this reason it is essential that the alkalies be included in the analysis, and the following scheme is so arranged as to include them:

Scheme for Water Analysis for Scale-Forming Ingredients.

Evaporate one liter of the water in a weighed platinum capsule, upon a water-bath to dryness ;[1] transfer to a hot-air-bath and heat at 105° C. for thirty minutes ; cool and weigh. Ignite slowly to a dull red heat until all carbonaceous matter is consumed ; cool and weigh. The loss of weight equals organic and volatile matter. Warm the contents of the capsule with ten to fifteen cc. hydrochloric acid, and twenty-five cc. water, boil and filter through an ashless filter into a 100 cc. graduated flask ; wash thoroughly, make contents of flask to containing mark with water ; mix well.

<table>
<tr>
<td colspan="3">(1) Residue.
Consists of insoluble mineral matter — SiO_2 or SiO_2, Al_2O_3. ($CaSO_4$.) It should be dried, ignited, and weighed : then fused with Na_2CO_3 in platinum crucible dissolved in H_2O, made acid with HCl, evaporated to dryness, taken up with $H_2O + HCl$, and filtered.</td>
<td colspan="6">(2) Solution.
100 cc. Divide into two portions, one of seventy-five cc. for bases and of twenty-five cc. for SO_3. 75 cc. Make alkaline with NH_4OH, boil and filter (all weights obtained to be divided by 3 and multiplied by 4.)</td>
<td rowspan="5">CO_2.
Is found by combining the chlorine and sulphuric acid with the bases, then determining how much CO_2 is required to convert the rest of the CaO and MgO to carbonates, as shown in the example given.</td>
<td rowspan="5">Cl.
Concentrate 250 cc. of the water in a porcelain dish to about 50 cc. ; add a few drops of K_2CrO_4 solution and titrate with a standard solution of $AgNO_3$, in which each cc. corresponds to 0.0017 gram chlorine.
Multiply the result by 4 = milligrams of Cl per liter.</td>
</tr>
<tr>
<td rowspan="4">Residue.
SiO_2 ; ignite and weigh as SiO_2.</td>
<td colspan="2">Filtrate.
Make alkaline with NH_4OH, boil, and filter.</td>
<td rowspan="4">(3) Residue
Consists of $Al_2O_3Fe_2O_3$ dry, ignite, and weigh as such.</td>
<td colspan="4">(4) Filtrate.
Add solution of ammonium oxalate ; set aside three hours, then filter.</td>
<td rowspan="4">SO_3.
25 cc. :
Warm, add solution of barium chloride, and allow to settle 3 hours ; filter, wash, dry, ignite, and weigh as $BaSO_4$. Calculate to SO_3 and multiply result by 4.</td>
</tr>
<tr>
<td rowspan="3">Residue.
$Al_2O_3Fe_2O_3$, dry, ignite, and weigh as such.</td>
<td rowspan="3">Filtrate.
Add solution of ammonium oxalate, set aside three hours, filter, dry, ignite, and weigh as CaO. Calculate to $CaSO_4$.</td>
<td rowspan="3">(5) Residue
Consists of CaC_2O_4. Dry, ignite, and weigh as CaO.</td>
<td colspan="3">(6) Filtrate.
Evaporate to dryness in platinum dish ; ignite to expel all ammonium salts ; cool, add water, boil and filter ; wash well.</td>
</tr>
<tr>
<td rowspan="2">(7) Residue
Consists of MgO,HO. Dry, ignite and weigh as MgO.</td>
<td colspan="2">(8) Filtrate.
Transfer to a weighed platinum dish, add a few drops of H_2SO_4, evaporate to dryness, and ignite to constant weight. This residue is composed of Na_2SO_4, $MgSO_4$, (K_2SO_4) ; after weighing dissolve in H_2O, make solution up to 50 cc. ; mix thoroughly and divide into two equal portions of 25 cc. each.</td>
</tr>
<tr>
<td>(1.) 25 cc. : Add a few drops of HCl, make alkaline with NH_4OH; then add, with constant stirring, solution of Na_2HPO_4 ; set aside 3 hours, filter, dry, ignite, and weigh as $Mg_2P_2O_7$; calculate this to $MgSO_4$, and subtract, after multiplying by 2, from total weight of sulphates in (8), and then the $MgSO_4$ is calculated to MgO.</td>
<td>(2). 25 cc.: Add a few drops of HCl, then solution of $PtCl_4$; evaporate on water-bath with some alcohol.[2] Filter off the K_2PtCl_6 on small tared filters, dry and weigh as K_2PtCl_6. Calculate this weight to K_2SO_4, and subtract, after multiplying by 2, from weight of Na_2SO_4 and K_2SO_4, and the residue left will be the weight of Na_2SO_4. The K_2SO_4 and Na_2SO_4 are then calculated to K_2O and Na_2O.</td>
</tr>
<tr>
<td>SiO_2.</td>
<td>(Insol.)
Al_2O_3,Fe_2O_3.</td>
<td>(Insol.)
$CaSO_4$.</td>
<td>(Sol.)
Al_2O_3 Fe_2O_3.</td>
<td>CaO.</td>
<td>MgO.</td>
<td></td>
<td></td>
<td>SO_3.</td>
<td>CO_2.</td>
<td>Cl.</td>
</tr>
<tr>
<td>↓</td><td>↓</td><td>↓</td><td>↓</td><td>↓</td><td>↓</td><td>↓</td><td>↓</td><td>↓</td><td>↓</td><td>↓</td>
</tr>
</table>

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[1] The water-bath, with constant level as shown in Figure 10, is to be recommended.

[2] Consult Fresenius Quant. Anal., p. 217, § 97.

Fig. 10.

To show, in detail, the method of using the scheme, the following water analysis is given. (Preliminary tests having shown the water to contain but little residue, eight liters of it were evaporated.)

Platinum capsule and residue (8 liters)	147.460	grams.
" " without residue	146.620	"
Total residue	0.840	"
Before ignition, capsule and residue	147.460	"
After " " " "	147.197	"
Organic, volatile, CO_2, etc	0.263	"
Crucible + SiO_2	15.970	"
Crucible	15.904	"
SiO_2	0.066	"

Solution made to 100 cc.—seventy-five cc. for bases, twenty-five cc. for SO_3.

Twenty-five cc. (SO_3).

Crucible and $BaSO_4$	16.023	grams.
Crucible	15.903	"
$BaSO_4$	0.120	"

Seventy-five cc.

Crucible + $Fe_2O_3(Al_2O_3)$	15.9338	grams.
Crucible	15.903	“
Fe_2O_3 (Al_2O_3)	0.0308	“
Crucible + CaO	16.0197	“
Crucible	15.903	“
CaO	0.1167	“
Crucible + MgO	15.928	“
Crucible	15.903	“
MgO	0.025	“
Platinum dish + alkaline sulphates + $MgSO_4$	53.370	“
Platinum dish	53.197	“
Sulphates	0.173	“

Dissolved in water, made solution up to fifty cc.: twenty-five cc, for magnesia determination, and twenty-five cc. for potash determination.

Crucible + $Mg_2P_2O_7$	15.942	grams.
Crucible	15.904	“
$Mg_2P_2O_7$	0.038	“

$$0.038 \times 2 = 0.076\ Mg_2P_2O_7.$$
$$Mg_2P_2O_7 : (MgSO_4)_2 :: 0.076 : x$$
$$MgSO_4 = 0.082 \text{ gram.}$$

Potassium platinic-chloride on tared filters = 0.023 gram, corresponds to 0.0046 potassium sulphate in the fifty cc.

Having determined the amounts of magnesium and potassium sulphates, the residue remaining is sodium sulphate, as follows:

Total sulphates	0.173	grams.
Magnesium sulphate	0.082	“
Sodium and potassium sulphates	0.091	“
Potassium sulphate	0.0046	“
Sodium sulphate	0.0864	“

and calculated to their oxides would be as follows:

MgO = 0.027 grams for 75 cc. = 0.036 gram for 100 cc. or the 8 liters.
Na_2O = 0.0377 “ “ 75 “ = 0.0502 “ “ “ “ “
K_2O = 0.0025 “ “ 75 “ = 0.0034 “ “ “ “ “

The chlorine found by titration amounted to 0.0055 gram per liter or 0.32 grain per gallon.

The weights thus obtained are in terms of the total residue, eight liters, and are converted into values corresponding to one liter, the result being as follows :

SiO_2	0.0082	gram per liter.
SO_3	0.0206	" " "
Cl	0.0055	" " "
K_2O	0.0005	" " "
Na_2O	0.0062	" " "
MgO	0.0077	" " "
CaO	0.0194	" " "
$Fe_2O_3(Al_2O_3)$	0.0051	" " "
CO_2	0.0137	" " "
Organic, etc	0.0193	" " "
	0.1062	" " "
Oxygen in excess of Cl	0.0016	" " "
Total residue	0.1046	" " "

It is necessary now to convert these values, grams per liter, into grains per gallon, in doing which the following table is used:

TABLE SHOWING THE NUMBER OF GRAINS PER U. S. GALLON (58,318 GRAINS) AND IMPERIAL GALLON (70,000 GRAINS) CORRESPONDING TO MILLIGRAMS PER LITER.

Milligrams per liter.	Grains per Imperial gallon.	Grains per U. S. gallon.
1	0.0700	0.0583
2	0.1400	0.1166
3	0.2100	0.1749
4	0.2800	0.2332
5	0.3500	0.2915
6	0.4200	0.3499
7	0.4900	0.4082
8	0.5600	0.4665
9	0.6300	0.5248
10	0.7000	0.5831
11	0.7700	0.6414
12	0.8400	0.6998
13	0.9100	0.7581
14	0.9800	0.8165
15	1.0500	0.8747
16	1.1200	0.9330
17	1.1900	0.9914
18	1.2600	1.0497
19	1.3300	1.1080
20	1.4000	1.1663

Milligrams per liter.	Grains per Imperial gallon.	Grains per U. S. gallon.	Milligrams per liter.	Grains per Imperial gallon.	Grains per U. S. gallon.
21	1.4700	1.2246	61	4.2700	3.5573
22	1.5400	1.2829	62	4.3400	3.6157
23	1.6100	1.3413	63	4.4100	3.6740
24	1.6800	1.3996	64	4.4800	3.7323
25	1.7500	1.4579	65	4.5500	3.7906
26	1.8200	1.5162	66	4.6200	3.8489
27	1.8900	1.5745	67	4.6900	3.9073
28	1.9600	1.6329	68	4.7600	3.9656
29	2.0300	1.6912	69	4.8300	4.0239
30	2.1000	1.7495	**70**	4.9000	4.0822
31	2.1700	1.8078	71	4.9700	4.1405
32	2.2400	1.8661	72	5.0400	4.1988
33	2.3100	1.9244	73	5.1100	4.2572
34	2.3800	1.9828	74	5.1800	4.3155
35	2.4500	2.0411	75	5.2500	4.3738
36	2.5200	2.0994	76	5.3200	4.4321
37	2.5900	2.1577	77	5.3900	4.4904
38	2.6600	2.2160	78	5.4600	4.5488
39	2.7300	2.2745	79	5.5300	4.6071
40	2.8000	2.3327	**80**	5.6000	4.6654
41	2.8700	2.3910	81	5.6700	4.7237
42	2.9400	2.4493	82	5.7400	4.7820
43	3.0100	2.5076	83	5.8100	4.8403
44	3.0800	2.5659	84	5.8800	4.8987
45	3.1500	2.6243	85	5.9500	4.9570
46	3.2200	2.6826	86	6.0200	5.0153
47	3.2900	2.7409	87	6.0900	5.0736
48	3.3600	2.7992	88	6.1600	5.1319
49	3.4300	2.8575	89	6.2300	5.1903
50	3.5000	2.9159	**90**	6.3000	5.2486
51	3.5700	2.9742	91	6.3700	5.3069
52	3.6400	3.0325	92	6.4400	5.3652
53	3.7100	3.0908	93	6.5100	5.4235
54	3.7800	3.1491	94	6.5800	5.4818
55	3.8500	3.2074	95	6.6500	5.5402
56	3.9200	3.2658	96	6.7200	5.5985
57	3.9900	3.3241	97	6.7900	5.6568
58	4.0600	3.3824	98	6.8600	5.7151
59	4.1300	3.4407	99	6.9300	5.7734
60	4.2000	3.4990	**100**	7.0000	5.8318

The amounts obtained being:

SiO_2	0.4771	grains per gallon.
SO_3	1.2012	" " "
Cl	0.3206	" " "
K_2O	0.0291	" " "
Na_2O	0.3615	" " "
MgO	0.4490	" " "
CaO	1.1313	" " "
CO_2	0.7987	" " "
Fe_2O_3,Al_2O_3	0.2973	" " "
Organic	1.1254	" " "
	6.1912	" " "
Oxygen in excess of Cl	0.0932	" " "
Total	6.0980	" " "

Having determined the component parts of the water residue in grains per gallon, it becomes necessary to unite these in chemical union, as near as possible, as they exist in the water.

The general rule may be stated as follows: The chlorine is combined with the sodium, if still in excess, then with the potassium, magnesium, and finally calcium. The sulphuric acid to the alkalies, provided there is not enough chlorine to saturate them, then to the calcium, and finally to the magnesium.

The carbon dioxide is united with the calcium and magnesium after the other combinations are made. There are exceptions to this rule, mineral waters and many artesian well waters forming notable examples.

Carrying out the above, the following is obtained:

	Gram per liter.	Grains per gallon.
NaCl	0.0091	0.5306
Na_2SO_4	0.0033	0.1923
K_2SO_4	0.0009	0.0524
$CaSO_4$	0.0311	1.8136
$CaCO_3$	0.0118	0.6880
$MgCO_3$	0.0162	0.9446
Fe_2O_3,Al_2O_3	0.0051	0.2973
SiO_2	0.0082	0.4771
Organic, etc	0.0193	1.1254
Total	0.1050	6.1213

This analysis shows that the principal scale-forming ingredient is calcium sulphate, being more than equal to the calcium and magnesium carbonates.

The following analysis is of a water containing sulphuric acid, but the alkalies being present in sufficient amount to combine with all of it, as well as the chlorine, no calcium sulphate is present:

	Gram per liter.	Grains per gallon.
SiO_2	0.0038	0.2215
SO_3	0.0110	0.6414
Cl	0.0062	0.3615
K_2O	0.0033	0.1923
Na_2O	0.0185	1.0788
MgO	0.0165	0.9624
CaO	0.0466	2.7175
Al_2O_3,Fe_2O_3	0.0020	0.1166
CO_2	0.0530	3.0908
Organic	0.0246	1.4345
	0.1855	10.8173
Oxygen in excess of Cl	0.0021	0.1224
Total	0.1834	10.6949

Combined as follows:

	Gram per liter.	Grains per gallon.
NaCl	0.0154	0.8980
Na_2SO_4	0.0141	0.8223
K_2SO_4	0.0061	0.3557
$CaCO_3$	0.0833	4.8577
$MgCO_3$	0.0338	1.9710
Al_2O_3,Fe_2O_3	0.0020	0.1166
SiO_2	0.0038	0.2215
Organic	0.0246	1.4345
Total	0.1831	10.6773

Where all the chlorine is not in combination with the sodium and potassium, magnesium chloride is usually present.

The latter compound, while not scale-forming, is considered as an active, corrosive agent—upon the supposition that at the temperature of 100° C., and higher, it is decomposed, and hydrochloric acid formed and liberated. Consult *Journal Society of*

Chemical Industry, Vol. IX, p. 472; also *Treatise on Steam Boilers*, by Wilson, p. 168.

The report, given below, is of a water from a driven well in Florida. Complaint having been made that not only was the scale excessive in amount, but that corrosive action was also very marked, an analysis was made; reference to which readily explains the difficulty encountered in the boilers.

	Gram per liter.	Grains per gallon.
NaCl	0.323	18.83
KCl	0.067	3.90
$MgCl_2$	0.104	6.06
$CaSO_4$	0.197	11.48
$CaCO_3$	0.293	17.08
$MgCO_3$	0.144	8.40
SiO_2	0.011	0.64
Al_2O_3, Fe_2O_3	0.007	0.40
Organic	0.138	8.04
Total	1.284	74.83

In all of the above analyses the constituents have been stated in grains per gallon, rather than in parts per 100,000, the former being in general use by the mechanical profession as the proper method by which to express the weights of the component parts of the residue of a water.

The following is an analysis of boiler water, in which no scale was present, but where corrosion was rapid. Sample was marked "Stand Pipe in Boiler."

NaCl	33.70	grains per gallon.
KCl	2.26	" " "
Na_2SO_4	16.33	" " "
$MgSO_4$	19.26	" " "
Fe_2O_3(suspended particles)	5.64	" " "
$Fe_2(NO_3)_2$	6.12	" " "
$Cu(NO_3)_2$	3.18	" " "
$Ca(NO_3)_2$	12.11	" " "
$Mg(NO_3)_2$	14.08	" " "
Silica	14.16	" " "
HNO_3(free)	12.27	" " "
Organic matter	24.12	" " "
Undetermined	2.15	" " "
Total	164.38	" " "

The water supplied to this boiler, also acid, was composed as follows:

NaCl	0.73	grains	per	gallon.
$MgCl_2$(KCl)	0.87	"	"	"
$MgSO_4$	1.71	"	"	"
$CaSO_4$	1.53	"	"	"
$Ca(NO_3)_2$	0.38	"	"	"
SiO_2	0.52	"	"	"
$Fe_2(NO_3)_3$	0.44	"	"	"
HNO_3(Free)	0.90	"	"	"
Organic matter	0.64	"	"	"
Total solids	7.72	"	"	"

This increase of total solids from 7.72 grains per gallon in the supply water to 164 grains total solids, per gallon of water in the boiler shows neglect in the management of the boilers.

By neutralizing the free acid, in the supply water, with sodium carbonate, corrosive action in the boiler was prevented.

In coal-bearing districts the boiler waters, while usually selected with care regarding the total solids, often contain free sulphuric acid, derived from the oxidation of the iron pyrites in the coal beds and which enter into the water supply.

The free acid in water can be determined quantitatively as follows:

250 cc. of the water are transferred to a six-inch porcelain evaporating dish, a few drops of litmus solution added, and the water boiled five minutes, then titrated with a very dilute standard solution of caustic soda.

Thus:

250 cc. of water taken, which required one and two-tenths cc. of the caustic soda solution.

The caustic soda solution is of such a strength that 31.1 cc. of it neutralizes five cc. of normal sulphuric acid solution.

One cc. of the normal sulphuric acid solution contains 0.049 gram sulphuric acid.

Then one cc. of the dilute caustic soda solution corresponds to 0.00787 gram sulphuric acid.

If 250 cc. of the water required one and two-tenths cc. of the caustic soda solution, one liter will require four and eight-tenths cc. caustic soda solution = 0.0377 gram sulphuric acid, corresponding to 2.20 grains of free sulphuric acid per gallon of water.

Determination of the Hardness of Water.

The hardness of water may be temporary, permanent, or both. The former is caused by calcium and magnesium carbonates and which are held in solution by the excess of carbon dioxide in the water.

Boiling the water drives out the excess of the carbon dioxide and the calcium and magnesium carbonates are thereby precipitated.

The permanent hardness is usually caused by calcium sulphate, which is not precipitated by boiling, or by magnesium chloride. The former, however, can be separated out of boiler-feed water by heating to 240° F. since at 230° F. it is insoluble.

Temporary Hardness.

The temporary hardness is determined as follows: Standard sulphuric acid solution and standard sodium carbonate solution are required and are prepared as follows:

1.96 grams of pure ignited sodium carbonate are dissolved in one liter of distilled water. One cc.=0.00106 gram corresponding to 0.001 calcium carbonate. The standard sulphuric acid solution is made of such strength that one cc. of it exactly neutralizes one cc. of the standard sodium carbonate solution.

100 cc. of the water, to which a few drops of lacmoid solution[1] have been added, are heated to boiling, and the sulphuric acid gradually added until the color changes.

Each cc. used represents one part of calcium carbonate per 100000 parts of water, or if it be desired to express it in grains per gallon, the result in parts per 100000 is multiplied by 0.583.

Permanent Hardness.

One hundred cc. of the water are taken and an excess of the sodium carbonate is added thereto, generally speaking the same volume will be sufficient. This is evaporated to dryness in a platinum dish, and the soluble portions are extracted with distilled water through a small filter and the filtrate is titrated with

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[1] Made by dissolving 2 grams lacmoid in 1000 cc. of dilute alcohol (50 %).

the standard acid for the excess of sodium carbonate; the difference represents the permanent hardness.

Reference.—Consult Sutton's volumetric analysis, p. 67.

Determination of Hardness by the Soap-Test.—(Phillips.)

The degree of hardness of a water is determined by ascertaining the amount of standard soap solution necessary to form a permanent lather with a definite volume of the sample; the "harder" the water the more soap it will consume, owing to the formation of insoluble calcium, magnesium, etc., soaps ("curd"), brought about by the decomposition of the soda or potash soap added, by the salts of the alkaline earths present in the water.

The hardness of water is usually expressed in terms of calcium carbonate.

Preparation of the standard solutions:

First, Solution of "Hard Water." Dissolve 1.11 grams of pure fused calcium chloride in a little water, and dilute to one liter at 15°C., or dissolve one gram of pure calcium carbonate in fifty cc. of dilute hydrochloric acid, evaporate to dryness, dissolve in fifty cc. of water, and dilute to one liter. In either case each cubic centimeter of the solution will correspond to 0.001 gram calcium carbonate.

Second, Solution of Soap. Castile soap, which is supposed to be made with soda and olive oil, is much used for standard soap solutions, but it has been found liable to considerable deterioration on keeping, especially in cold weather, owing to the deposition of sodium palmitate.

Sodium oleate makes a standard soap solution which suffers very little change on keeping, and can be generally recommended for the purpose.

Thirteen grams of it are dissolved in a mixture of 500 cc. of alcohol and 500 cc. of water, and filtered if necessary. It now becomes necessary to standardize it, so that one cc. will be equivalent to 0.001 gram of calcium carbonate. In order to effect this, twelve cc. of the standard hard water are run into a 250 cc. stoppered bottle from a burette and diluted to 58.3 cc. A burette is now filled with the soap solution which is run into the bottle one cc. at a time, and the bottle vigorously shaken after each addition, until a point is reached where a persistent lather, lasting for at least five minutes, is obtained. Note the volume required. Twelve cc. of hard water should require thirteen cc. of soap solution (distilled water itself requiring one cc. to form a lather), but it will be a figure less than this, and therefore the soap solution is too strong and will require diluting, so that twelve cc. of standard "hard" water will require thirteen cc. of the soap solution. An example of an actual preparation of a standard soap solution will explain this.

Thirteen grams of sodium oleate were dissolved in a mixture of 500 cc. of alcohol and 500 cc. of water, and filtered. On testing in the manner described, twelve cc. of the standard "hard" water diluted to 58.3 cc. required 11.4 cc. of the soap solution to form a persistent lather.

Now, since thirteen cc. should have been required, every 11.4 cc. of the soap solution left, requires diluting by $13 - 11.4 = 1.6$ cc.

There were 960 cc. of the solution left, therefore $\frac{960}{11.4} = 84.2$, and $84.2 \times 1.6 = 134.7$ cc. more of the mixture of alcohol and water to be added. On adding this quantity, thoroughly mixing, and testing as before, twelve cc. of the standard hard water required exactly thirteen cc. of the soap solution.

Determination of Total Hardness.

58.3 cc.[1] of the clear sample, of the water to be examined, are run into a 250 cc. flask, and the standard soap solution added in the manner described above, until a lather capable of persisting for five minutes is produced. The number of cubic centimeters required, minus one cc. for the water itself, will give the degrees of hardness in terms of calcium carbonate in grains per gallon. If the water contains a fair proportion of magnesia salts, there will be some difficulty in obtaining the right point, owing to the slowness with which magnesia salts decompose soap ; an appar-

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[1] If it be desired to determine the hardness in grains per English Imperial gallon, instead of the United States gallon, seventy cc. of the water must be taken. This is dependent upon the fact that the English Imperial gallon contains 70,000 grains, and the United States gallon 58,318 grains.

ent persistent lather is formed, which on being allowed to stand a little while and again shaken up, will disappear; a little experience with magnesian hard waters will familiarize the operator with this peculiarity.

The Permanent Hardness.

250 cc. of the water are poured into a 500 cc. flask, and boiled for one-half hour, the original volume being kept up by frequent additions of boiling distilled water, free from carbon dioxide. After cooling, it is quickly poured into a 250 cc. graduated stoppered flask, diluted if necessary to exactly 250 cc. at 15° C. with distilled water, well mixed and filtered. 58.3 cc. of the solution are now poured into the bottle and the permanent hardness determined as described.

The Temporary Hardness.

The temporary hardness, or that hardness removed by boiling, is obtained by deducting the degree of permanent hardness from that of the total.

Standards of Hardness.

The French standard of hardness of water is stated in terms of milligrams of calcium carbonate in 100 grams of water, or, parts calcium carbonate per 100,000 parts of water.

The German standard represents milligrams of lime in 100 grams of water, or parts lime per 100,000 parts of water.

The English standard represents grains of calcium carbonate per gallon of 70,000 grains.

The American standard represents grains of calcium carbonate per gallon of 58,381 grains. The French standard is to be preferred.

TABLE SHOWING THE RELATIVE HARDNESS OF THE WATER SUPPLIED TO CITIES. DETERMINATIONS MADE BY A. R. LEEDS.

Calcium carbonate.	Philadelphia.	New York.	Brooklyn.	Jersey City.	Boston.	Washington.	Rochester.	Cincinnati.
Parts per 100,000......	4.4	3.3	2.2	3.2	2.1	4.8	5.5	6.4
Grains per U. S. gallon	2.56	1.92	1.28	1.86	1.22	2.79	3.20	3.73

The Sanitary Analysis of Water.

This comprises the determination of

1. Chlorine.
2. Free and albuminoid ammonia.
3. Nitrates.
4. Nitrites.
5. Total solids.
6. Organic and volatile matter by ignition of residue.
7. Oxygen required to oxidize organic matter.

1. Determination of Chlorine. Standard Silver Solution.

Dissolve five grams of pure crystallized silver nitrate in 1000 cc. of distilled water. One cc. of the solution is equivalent to 0.001 gram chlorine. If the water to be tested shows by qualitative analysis a small amount of chloride present, 250 cc. of the water should be evaporated to about fifty cc., allowed to cool, three drops of saturated solution of potassium chromate added, and the silver nitrate solution dropped carefully from a burette until a faint permanent red color is produced in the water. This point indicates that all the chlorine has combined with the silver, and that any additional silver solution added forms silver chromate. Thus:

250 cc. of the water used for examination.
" " " " required 1.3 cc. silver nitrate solution.
1000 cc. " " " " 5.2 cc. " " "
Equivalent to 0.0052 grams of chlorine per liter.
" " 0.52 parts chlorine in 100,000 parts of the water.
" " 5.20 " " " 1,000,000 " " " "

It is customary to state the amount of chlorine as "chlorine as chlorids"—as $NaCl$. Thus:

0.0052 gram chlorine per liter = 0.0085 gram sodium chloride per liter.
0.52 parts chlorine per 100,000 = 0.85 parts sodium chloride per 100,000.
5.2 " " " 1,000,000 = 8.5 " " " " 1,000.000.

The amount of chlorine allowable in good drinking water cannot be stated positively, since the source from which it is derived must be taken into account.

Results from a great many analyses of various waters would indicate the amount allowed as follows :

Rain water	Traces to one part per 1,000,000.
Surface water	One to ten parts per 1,000,000.
Subsoil	Two to twelve parts per 1,000,000.
Deep well water	Traces to large quantity.

2. *Free and Albuminoid Ammonia.*

Solutions required are :

a. Standard solution of ammonium chloride, made by dissolving 0.382 gram dry ammonium chloride in 100 cc. of ammonia-free distilled water. One cc. of this solution is diluted to 100 cc. with distilled water, each cc. of the latter solution corresponding to 0.000012 gram ammonia.

b. Standard Nessler Reagent.—Dissolve seventeen grams of mercuric chloride (pulverized) in 300 cc. of water, and thirty-five grams of potassium iodide in 100 cc. of water. Pour the mercuric chloride solution into the potassium iodide until a permanent red precipitate is formed. Add a twenty per cent. solution of sodium hydroxide until the volume of the mixed solution amounts to one liter. Add some more mercuric chloride solution until a permanent red precipitate forms and allow to settle.

c. Alkaline potassium permanganate, formed by dissolving eight grams of potassium permanganate and 200 grams of potassium hydroxide in a liter of distilled water.

This solution is concentrated by boiling to about 750 cc., then 250 cc. of ammonia-free water is added. When properly prepared this solution gives but traces of ammonia by distillation. In any event, however, it must be tested, and if an appreciable amount is found, it must be deducted from the determination of albuminoid ammonia in any sample of water under examination.

Ammonia-free water is made by distilling water acidulated with sulphuric acid.

Process.

The apparatus shown in Fig. 11 is well adapted for this purpose.

Place 250 cc. of the water to be tested in a flask, capacity

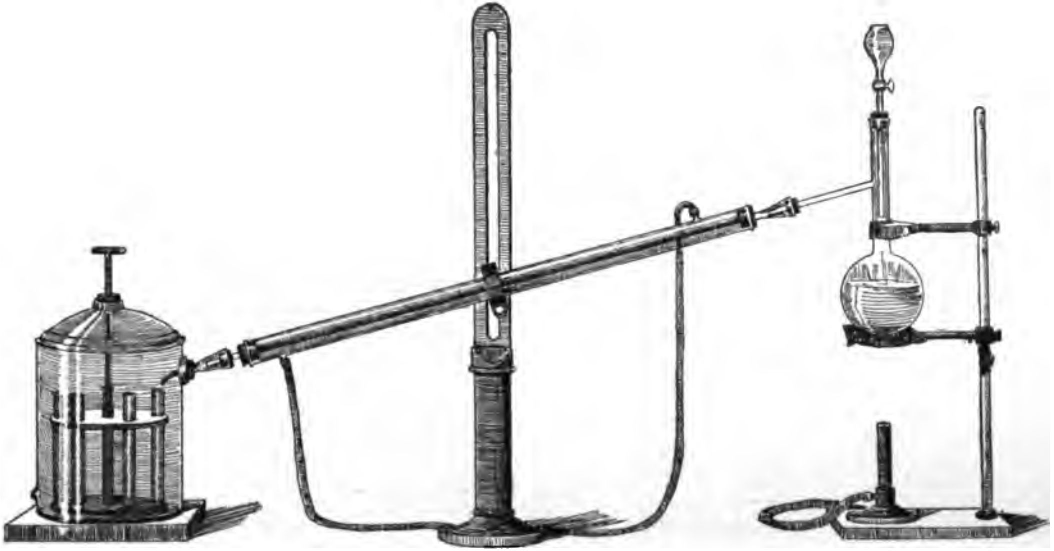

Fig. 11.[1]

[1] The delivery tube in distilling flask is fastened to and passes through the ground-glass stopper.

one liter, add one cc. saturated solution sodium carbonate, connect with the condenser and distil until no reaction for ammonia is shown in the distillate, (caught in one of the comparison tubes)[1] when two cc. of the Nessler solution is added thereto, a yellowish brown color being indicative of ammonia. The apparatus being free from ammonia, 500 cc. of the water are now added to the water remaining in the flask and one cc. of the saturated sodium carbonate solution (free from ammonia) added. Distillation proceeds until three distillates, each of fifty cc., have been received in the comparison tubes, when the distillation is stopped and the heat removed until the distillates can be examined. The comparison tubes are protected by being enclosed in a glass vessel, with a movable top, as shown in Fig. 11, at the base of which is an opening filled with cotton wool.

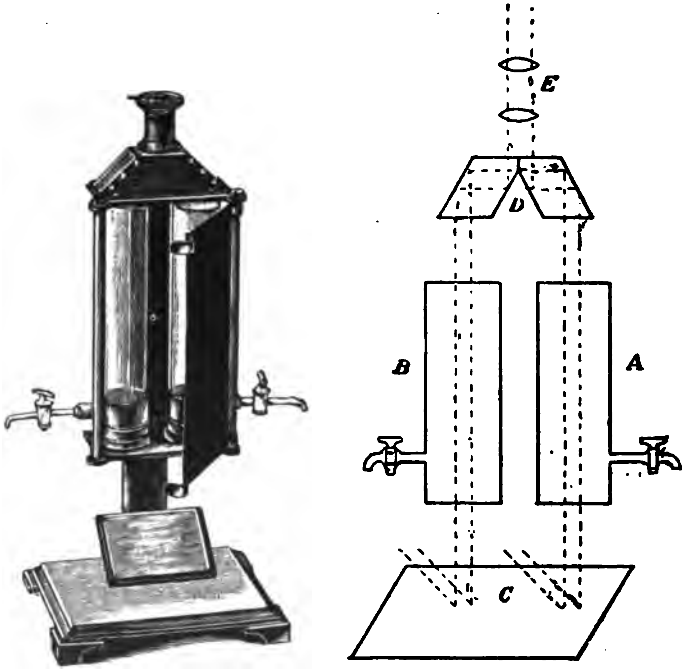

Fig. 12.

These comparitor tubes have a mark indicating fifty cc., and

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[1] See Fig. 11.

when the distillate reaches that mark, the handle of the stand containing the comparitor tubes is turned and another comparitor tube placed under the outlet of the condenser. The revolving stand contains seven comparitor tubes, sufficient for both the free and albuminoid ammonia determinations. C. H. Wolff's colorimeter, Fig. 12, has an extended use in water analysis for the purpose of comparing tints of color of the water, also in the determination of the difference in color in the estimation of free and albuminoid ammonia.

One of the tubes contains the nesslerized standard ammonium chloride solution, the other tube a portion of the water distillate, nesslerized, to compare with the former. The contents of the tube containing the darker liquid are partially drawn off by means of the glass stop-cock at the base, and the remaining liquid diluted with distilled water until a uniform tint of color is obtained in both glasses. As these tubes are graduated, the calculations are simplified and rendered more expeditious.

Ammonia Determinations.

The first fifty cc. of distillate is now tested for ammonia, as follows:

The tube is removed and placed in a comparitor and two cc. of the Nessler solution added. The color produced must be matched by taking another tube and filling to the fifty cc. mark with ammonia-free distilled water, adding two cc. Nessler solution and one cc. of the standard ammonium chloride solution. Allow to stand five minutes for full development of color, then compare the color of the liquids in the tubes.

If the solution containing the ammonium chloride is too strong, divide it and add distilled ammonia-free water to fifty cc. mark and compare again, and repeat until the tints are identical.

If, however, the solution containing the ammonium chloride is not deep enough in color, add one cc. more of the standard ammonium chloride solution and compare as before.

The second and third distillate are treated in a similar manner, but if the third distillate shows over a trace of ammonia, a fourth distillate must be taken, or until no appreciable amount of free ammonia can be obtained.

Free Ammonia.

500 cc. of the water taken.

First	distillate	(50 cc.)	required	1.5 cc.	ammonium	chloride	solution.
Second	"	(50 cc.)	"	0.3 cc.	"	"	"
Third	"	(50 cc.)	"	none	"	"	"
		Total for	500 cc.	1.8 cc.	"	"	"
		" "	1000 cc.	3.6 cc.	"	"	"

One cc. ammonium chloride solution is equivalent to 0.00001 gram nitrogen, or 0.000012 gram ammonia.

Then one liter of the water contains 0.000043 gram free ammonia.

Equivalent to 0.0043 part ammonia per 100,000.
" " 0.0430 " " " 1,000,000.

Fifty cc. of the alkaline solution potassium permanganate are added to the contents of the flask, after the determination of the free ammonia. The contents of the flask must be cooled somewhat before the addition of the alkaline permanganate solution. The latter is placed in the flask by means of the glass delivery tube, which passes through and is fused to the glass stopper of the flask. By this arrangement any solution can be added to the contents of the flask without removing the stopper.

The distillation and comparison of distillates by known amounts of ammonium chloride solution are made in the same manner as for the determination of free ammonia.

Albuminoid Ammonia.

750 cc. of the water taken.

First	distillate	required	3.2 cc.	ammonium	chloride	solution.
Second	"	"	0.7 cc.	"	"	"
	Total	"	3.9 cc.	"	"	"
	1000 cc.	"	5.2 cc.	"	"	"

Equivalent to 0.000063 gram ammonia per liter.
" " 0.0052 part ammonia per 100,000 parts.
" " 0.0520 " " " 1,000,000 "

It must be remembered that the free ammonia was determined in the 500 cc. of water after the free ammonia was expelled from the 250 cc. of water first placed in the flask.

As the albuminoid ammonia is not developed until the addition of the alkaline permanganate solution, the determination of the albuminoid would be upon 750 cc. of water, as above stated.

The amounts of free and albuminoid ammonia allowable in good drinking water are thus stated by Wanklyn: "If a water yield 0.00 part of albuminoid ammonia per million, it may be passed as organically pure, despite of much free ammonia and chlorides; and indeed if the albuminoid ammonia amounts to 0.02, or to less than 0.05 parts per million, the water belongs to the class of very pure water. When the albuminoid ammonia amounts to 0.05, then the proportion of free ammonia becomes an element in the calculation, and I should be inclined to regard with some suspicion a water yielding a considerable quantity of free ammonia along with more than 0.05 part of albuminoid ammonia per million. Free ammonia, however, being absent, or very small, a water should not be condemned unless the albuminoid ammonia reaches something like 0.10 part per million. Albuminoid ammonia above 0.10 per million begins to be a very suspicious

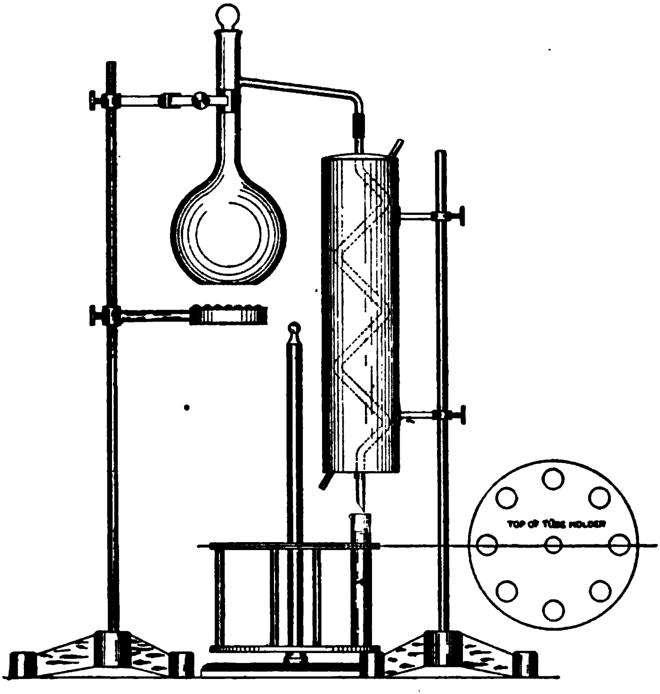

Fig. 13.

sign; and over 0.15 ought to condemn a water absolutely. The absence of chlorine or the absence of more than one grain of chlorine per gallon, is a sign that the organic impurity is of vegetable rather than of animal origin, but it would be a great mistake to allow water highly contaminated with vegetable matter to be taken for domestic use."

The apparatus for the determination of free and albuminoid ammonia, used by the New York City Health Department, is shown in Fig. 13, a description of which will be found in the *Journal of the American Chemical Society*, **16**, 871.

3. Determination of Nitrates by the Phenol Method.

a. Standard potassium nitrate solution, formed by dissolving 0.722 gram potassium nitrate, C. P., in a liter of water. One cc. of this solution is equivalent to 0.00044 NO_2.

b. Phenolsulphonic acid, formed by adding three cc. of water, six grams pure phenol and thirty-seven cc. of concentrated sulphuric acid together.

The operation of determining the nitrate is as follows:

Twenty-five cc. of the water are evaporated to dryness in a No. 2 porcelain capsule, on a water bath. One cc. of the phenolsulphonic acid is added and incorporated thoroughly with the residue.

Add one cc. water, three drops of concentrated sulphuric acid and warm. Dilute with twenty-five cc. water, make alkaline with ammonium hydroxide and make solution up to 100 cc. with water. If an appreciable amount of nitrate is present, it forms picric acid with the phenolsulphonic acid, imparting a yellow color to the solution, when the ammonia is added by the formation of ammonium picrate. The intensity of the color is proportional to the amount of ammonium picrate present.

One cc. of the standard potassium nitrate solution is evaporated in a porcelain capsule, treated as above, and the solution made up to 100 cc. The two solutions are placed in comparitor glass tubes and distilled water added to one or the other until the colors agree in tint. Suppose twenty-five cc. of the original water after treatment and subsequent dilution to 100 cc. corresponded in color to the standard solution of one cc., which after treatment and dilution to 100 cc. was diluted to 200 cc. Then twenty-five cc. of the original water contained 0.00005 gram nitrogen, or 1000 cc. contained 0.0020 gram nitrogen or 0.009 gram NO_3 per liter, corresponding to 0.52 grains per gallon, or 0.9 part per 100,000, or 9.0 parts per 1,000,000.

4. *Nitrites.*

Glosway's modification of Griess's method is to be recommended for simplicity and accuracy.

The solutions required are :

1. Sulphanilic acid. Dissolve one gram in 300 cc. of acetic acid (sp. gr. 1.04).

2. Sodium nitrite. Formed by dissolving 0.272 gram silver nitrite in 100 cc. water, adding a dilute solution of sodium chloride in slight excess, and diluting to 250 cc.

Take 100 cc. of this solution, dilute to one liter for use. One cc. = 0.00001 gram nitrogen.

3. *a*-amido-naphthalene acetate.

Two-tenths gram of naphthylamine is boiled with forty cc. of water, filtered and diluted to 400 cc.

Process of Determination.

Twenty-five cc. of water are taken and placed in one of the color comparitors, two cc. of the sulphanilic acid and two cc. of the amido-naphthalene acetate are added. If nitrites are present, a pink color is produced, which must be compared with the color produced by one cc. of the standard nitrite solution, to which two cc. of the sulphanilic acid, two cc. of the amido-naphthalene acetate and twenty-five cc. of pure distilled water (free from nitrites) are added.

Suppose twenty-five cc. of the water required, six-tenths cc. of the standard nitrite solution, or 0.000006 gram nitrogen, or 0.00002 gram NO_2.

Corresponding to 0.0008 gram NO_2 per liter.
" " 0.0466 grain per gallon.
" " 0.0800 part per 100.000.
" " 0.8000 part per 1,000,000.

5. The total solids are determined by evaporating 500 cc. of

the water in a platinum dish and drying the residue at 105° C. to constant weight. The amount obtained multiplied by 2 equals the weight per liter.

6. The organic and volatile matter is approximately determined by igniting the weighed residue until all carbonaceous matter is consumed, and weighing; the difference between the weight of the total solids and the weight after ignition is the volatile and combustible matter.

7. Oxygen required to oxidize the organic matter in the water.

.Solutions required:

Standard potassium permanganate, formed by dissolving 0.395 gram potassium permanganate in 1000 cc. water. Each cc. contains 0.0001 gram available oxygen.

Potassium Iodide Solution. 100 grams of the pure salt dissolved in 1000 cc. water.

Dilute Sulphuric Acid Solution. One part by volume of pure sulphuric acid is mixed with three parts by volume of distilled water and solution of potassium permanganate dropped in until the whole retains a *very faint* pink tint, after warming to 80° F. for four hours.

Sodium Thiosulphate. One part of the pure crystallized salt in 1000 parts of salt.

Starch Indicator is made by mixing six grams of starch with 100 cc. pure glycerine, heating for one hour to 100° C., pouring it into 200 cc. of water, then adding sufficient strong alcohol to precipitate the soluble starch, which is filtered off and preserved in a moist pasty state. When required, a minute quantity is taken with a glass rod.

Determination of the Oxygen Absorbed.

Two separate determinations are required, *viz*., the amount of oxygen absorbed during fifteen minutes and that absorbed during four hours. Both are to be made at a temperature of 27° C. It is most convenient to make these determinations in twelve ounce stoppered flasks, which have been rinsed with sulphuric acid and then with water. Put 250 cc. of the water to be tested into each flask, which must be immersed in a water bath or suitable air bath until the temperature rises to 27° C. Now add to each flask ten cc. of the dilute sulphuric acid, and then ten cc. of the standard permanganate solution. Fifteen minutes after the addition of the permanganate, one of the flasks must be removed from the bath and two or three drops of the solution of potassium iodide added to remove the pink color. After thorough admixture, run from a burette the standard solution of thiosulphate until the yellow color is nearly destroyed, add some of the starch solution and continue the addition of the thiosulphate until the blue color is just discharged. If the titration has been properly conducted, the addition of one drop of permanganate solution will restore the blue color. At the end of four hours remove the other flask, add potassium iodide and titrate with thiosulphate, as just described. Should the pink color of the water in the flask diminish rapidly during the four hours, further measured quantities of the standard solution of permanganate must be added from time to time so as to keep it markedly pink. The thiosulphate solution must be standardized, not only at first, but (since it is liable to change), from time to time in the following way: To 250 cc. of pure distilled water add two or three drops of the solution of potassium iodide, and ten cc. of standardized solution of permanganate. Titrate with thiosulphate solution as above described. The quantity used will be the amount of thiosulphate solution corresponding to ten cc. of the standardized permanganate, and the factor so found must be used in calculating the results of the thiosulphate titrations to show the amount of standard permanganate solution used, and thence the amount of oxygen absorbed. The amount of thiosulphate solution thus found to be required to combine with the iodine liberated by the permanganate.

CONVERSION TABLE.

Parts per	100,000	× 0.7	= Grains per Imperial Gallon.
" "	1,000,000	× 0.07	= " " " "
" "	100,000	× 0.583	= " " U. S. "
" "	1,000,000	× 0.058	= " " " "
" "	1,000,000	× 0.00833	= Avoir. pounds per 1000 U.S. Gal.
Grains "	Imp. gal.	÷ 0.7	= Parts per 100,000
" "	" "	÷ 0.07	= " " 1,000,000
" "	U. S. "	÷ 0.583	= " " 100,000
" "	" "	÷ 0.058	= " " 1,000,000

Analyses of American Waters.

COMPILED BY C. F. CHANDLER FROM THE REPORT OF PROF. J. W. MALLET, REPORT OF THE NATIONAL BOARD OF HEALTH FOR 1882, WASHINGTON, D. C.—PARTS BY WEIGHT IN ONE MILLION.

Mallet's number.	Character of the water.	Total solids at 100° C.	Loss on ignition.	Chlorine.	Frankland's combustion process.		Albuminoid ammonia process.		Nitrogen of nitrites. Griess's naphthylene test.	Nitrogen of nitrates. Crum's mercury method.	Permanganate process. Tidy's method, cold. Oxygen consumed.		Permanganate process. Kubel's method boiling. Oxygen consumed.	Dissolved oxygen.
					Organic carbon.	Organic nitrogen.	Free ammonia.	Albuminoid ammonia.			In 1 hr.	In 3 hrs	In 10 m	
1	Rain water, iron tank, New Orleans, La.	55	35	1.70	1.75	0.99	0.045	0.145	trace	0.500	1.522	1.964	2.091	5.84
5	Pure spring, University of Virginia	20	trace	0.75	0.36	0.08	0.020	0.020	none	trace	0.140	0.248	0.632	7.87
20	Public spring, Harrisonburg, Va	270	20	4.00	1.49	0.52	0.130	0.144	0.184	5.060	0.082	0.226	0.528	6.49
23	Lake Champlain, Burlington, Vt	70	50	0.70	2.81	0.32	0.035	0.140	none	trace	1.132	1.388	1.525	7.06
19	Lake Michigan, Chicago, Ill............	160	60	2.00	1.40	0.31	0.095	0.090	none	trace	0.264	0.400	1.500	9.08
13	Lake Roland, Baltimore, Md............	110	35	2.33	1.67	0.39	0.010	0.110	trace	0.370	0.520	0.684	1.328	4.25
11	Cochituate, Boston, Mass................	60	10	3.60	7.20	0.66	0.070	0.255	trace	trace	4.008	5.213	7.443	6.76
12	Croton, New York City..................	90	20	0.67	3.96	0.62	0.020	0.150	0.018	trace	1.799	2.163	3.433	6.67
44	Hudson River, Poughkeepsie, N. Y......	85	40	4.50	2.97	0.56	0.050	0.125	trace	trace	1.092	1.376	2.287	7.08
47	Crosswicks Creek, Bordentown, N. J....	80	45	2.33	3.07	0.53	0.035	0.175	0.005	trace	1.699	2.211	2.719	6.60
14	Schuylkill, Philadelphia, Pa............	130	30	6.00	2.19	0.65	0.105	0.210	trace	0.220	1.172	1.436	2.140	6.63
15	Potomac, Washington, D. C	165	95	1.10	2.20	0.44	0.050	0.127	trace	0.230	0.864	1.016	1.021	7.54
16	James River, Richmond, Va..............	105	20	1.17	1.41	0.33	0.550	0.150	trace	trace	0.512	0.636	1.654	6.41
26	Mississippi River, Rock Island, Ills.....	140	95	1.00	7.79	0.52	0.025	0.260	none	trace	3.340	4.188	6.000	7.46
17	Mississippi River, New Orleans, La.....	340	50	14.50	6.14	0.76	0.040	0 325	none	0.080	2.398	3.142	5.724	5.33
45	Trinity River, Dallas, Texas............	460	170	13.50	22.10	0.76	0.040	0.370	none	0.470	1.172	1.708	2.534	7.91
49	Oil Creek, Titusville, Pa.................	100	50	0.75	3.78	0.23	0.075	0.150	none	none	2.044	2.472	3.746	6.06
21	Driven wells, Titusville, Pa	200	30	4.00	0.99	0.34	0.030	0 025	0.055	1.130	1.093	1.181	none	7.81
35	Driven well, Asbury Park, N. J..........	225	50	66.00	0.94	0.40	0.358	0.058	0.033	8.507	0.262	0.266	0.107	7.81
9	Well, University of Virginia.............	40	30	3.75	0.38	0.20	0.015	0.020	trace	1.040	0.216	0.284	0.097	7.57
24	Well, Fairfax Court House, Va..........	60	45	2.00	0.68	0.26	0.070	0.070	0.037	1.123	0.278	0.410	none	6.64
42	Well, Ripley, Miss.........................	70	20	3.25	0.73	0.46	0.140	0.046	trace	trace	0.192	0.228	0.486	6.59
41	Well, Closter, N. J.....................	115	65	5.00	0.35	0.07	0.025	0.020	none	2.830	0.160	0.192	none	8.48
34	Well, Jacksonville, Fla	170	60	28.00	1.47	0.35	0.230	0.095	.018	4.342	0.467	0.807	1.049	6.99
40	Well, Port Chester, N. Y.................	200	80	29.00	2.17	1.41	0.025	0.120	0.092	7.038	0.111	0.271	0.485	7.32
30	Well, Jonesville, Mich....................	245	50	7.00	4.11	1.03	0.220	0.150	0.276	3.164	2.880	3.764	4.674	5.45
25	Well, Andover, Mass......................	260	100	46.60	0.73	0.53	0.010	0.045	none	6.790	0.104	0.104	none	7.18
37	Well, Meridian, Miss......................	260	100	56.00	0.67	0.26	0.015	0.070	0.004	8.496	0.132	0.300	0.288	7.21
28	Well, Wellsville, Ohio....................	320	130	20.03	0.97	0.77	0.070	0.100	trace	6.610	0.586	0.834	1.102	6.89
38	Well, Lexington, Mich.	370	20	18.70	3.75	0.73	0.080	0.150	0.011	3.239	1.407	1.843	2.737	6.52
31	Well, Adamstown, Md......................	420	120	41.50	0.86	1.28	0.140	0.240	0.040	8.740	0.934	1.478	0.586	6.01
32	Well, City Park, Charlotte, Mich	400	60	7.50	0.98	0.57	0.042	0.080	none	7.060	0.120	0.138	none	7.03
27	Well, Little Rock, Ark....................	610	320	93.00	0.86	6.88	0.190	0.075	0.037	28.403	0.126	0.126	0.639	6.31
50	Well, Newport, R. I........................	720	170	1.08	1.41	1.51	0.025	0.080	0.009	30.361	0.183	0.383	1.106	7.56
36	Well, Huntingdon, Pa	810	280	106.00	2.03	0.03	0.120	0.086	0.029	15.961	0 430	0.662	0.453	7.09
43	Well, Union City, Tenn...................	830	330	132.00	1.17	0.98	0.030	0.050	none	13.050	0.724	1.076	0.344	6.22
29	Well, Mount Vernon, Ohio...............	1160	400	79.00	0.50	0.55	0.150	0.100	none	24.990	0.324	0.404	0.535	7.11
2	Artesian well, Charleston, S. C..........	1170	100	130.00	1.57	0.59	0.300	0.040	0.368	none	0.527	0.927	2.043	5.85
53	Ice. Almy's Pond, Newport, R. I.........	65	40	1.33	1.27	0.33	0.080	0.155	trace	trace	0.292	0.388	0.973	6.84

Composition of European Waters.

Source.	Total solid contents. Parts in 1,000,000.	Constituents of solid contents; parts by weight in 1,000,000.													Gaseous contents cc. per liter.		
		Ca	Mg	Na	K	Al	Fe	Mn	CO_3	SO_4	Cl	SiO_2	NO_3	Organic.	CO_2	O	N
Thames, at London Bridge	408.4	82.1	..	14.3	1.7	..	..	..	69.4	32.2	63.6	1.8	..	100.0	27.2	..	..
Lake of Geneva	152.0	36.4	10.5	..	..	..	..	..	48.2	43.2	6.7	1.0	..	6.0	..	..	..
Rhone, at Lyons	184.0	65.9	1.4	..	..	..	..	..	90.0	19.7	..	7.0	..	..	..	..	..
Loire, at Orleans	134.6	19.2	1.7	9.3	..	7.1	5.5	..	41.5	2.3	2.9	40.6	..	..	1.8	..	..
Garonne, at Toulouse	136.7	25.8	0.9	5.8	3.4	..	3.1	3.0	44.8	7.8	1.9	40.1	..	..	17.0	7.9	15.7
Aar, **near Berne**	216.3	66.0	10.0	0.3	..	..	..	..	103.3	33.7	0.3	2.7	..	..	..	..	..
Rhine, at Basle	169.4	55.5	4.8	0.6	..	..	..	..	86.2	15.4	1.5	2.1	..	3.3	..	..	..
Elbe, near Hamburg	126.9	27.9	1.1	..	..	..	1.2	..	44.7	7.2	39.4	5.4	..	..	..	..	..
Spree, at Berlin	114.0	26.0	2.6	2 3	1.5	13.0	..	..	45.4	8.6	0.6	..	3.0	..	..	..	..
Danube, near Vienna	141.4	34.3	7.0	..	..	..	..	..	60.9	13.1	2.0	4 9	..	..	..	..	..

Analyses of Ocean Waters.

Source.	Parts by weight in 1,000,000 Total solid con'nts.	Constituents of solid contents; parts by weight in 1,000,000.						Constituents of solid contents; parts by weight in 1,000,000.							
		Ca	Mg	Na	K	NH_4	Al	Fe	Mn	CO_3	SO_4	Cl	Br	PO_4	SiO_2	Organic
Atlantic Ocean, 41° 18' N., 36° 28' W.	38400.0	556.8	1198.1	17719.7	668.2	....	....	....	....	....	3029.8	20839.7	387.8	....	....	......
Pacific Ocean, 28° 11' S., 93° 24' W., 420 feet deep	35200.0	475.2	1471.4	10232.6	633.6	....	....	....	....	....	2826.6	19321.3	239.4	....	....	......
German Ocean, between Belgium and England	32800.0	324.4	1158.2	10205.7	353.6	....	....	....	....	....	2590.2	18167.9	....	trace	16.3	......
Baltic	17710.0	36.3	611.5	5894.1	....	....	....	....	....	....	718.9	10386.4	....	....	....	......
Mediterranean, off Cette	37700.0	444.1	1310.4	11706.2	264.3	....	....	2.8	....	67.9	2942.8	20526.9	433.5	....	....	......
Black Sea, off southern coast of Crimea	1766.1	130.5	662.2	5512.0	97.5	....	....	127.1	....	247.5	1250.2	9574.5	5.0	....	....	......
Caspian Sea	6296.0	191.6	409.8	1444.0	139.7	....	40.0	....	. ..	77.3	1337.2	2737.7	....	....	....	......
Dead Sea	240483.3	9000.0	19883.4	47918.0	6385.1	18	153.4	11.7	25.8	....	479.0	154442.0	2176.7	....	. ..	617.3

Water supplied to large cities is usually filtered through sand filter beds.

Fig. 14 shows the section of a well-arranged filter bed built for the city of Dublin. The bottom of the filter is composed of puddled clay three-fifth meter in thickness **A**, built in with stones one-fifth meter in thickness. The next layer, three-quarters meter thick, consists of coarse angular stones **B**, then fifteen centimeters of smaller stones **C**, followed by a layer fifteen centimeters in depth of coarse gravel **D**, then the same depth of fine gravel **E**, and finally three-fourth meter of sand **F**. To collect the water there are two channels B, situated half in the bed of clay and half in the stratum of large stones. Each channel is seventy-five centimeters in width and sixty centimeters in depth. The surface of sand in each meter is sixty-one by thirty-one meters ; the depth of this water is sixty centimeters.

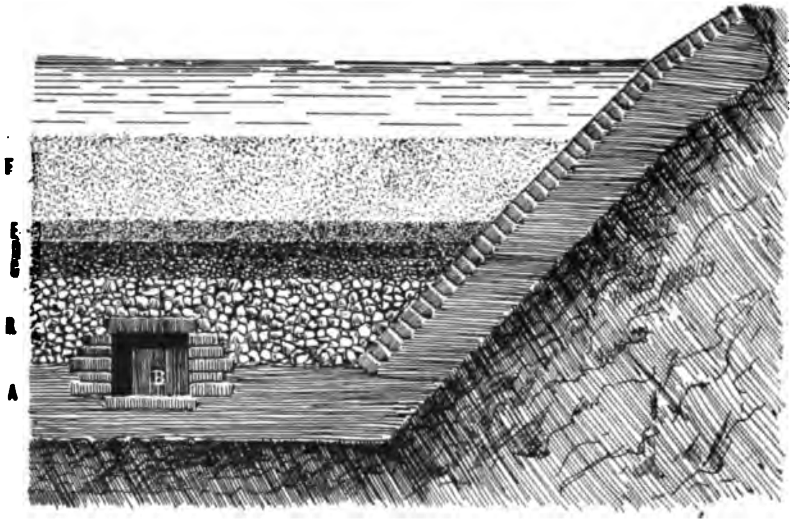

Fig. 14.

The speed of filtration varies in the existing sand filters from one and four-tenths to fifteen meters per twenty-four hours. Each water requires, if it is to be well filtered by a given sand, a determined speed of filtration. Thus, under otherwise similar conditions, three and five-tenths cubic meters of Thames water may be filtered in twenty-four hours per square meter of filtering surface, but only one and seven-tenths cubic meters of Elbe water, as the latter contains much more finely divided dirt.

In a well managed filtration the turbid water passes so slowly through the sand that each of the fine particles of dirt, though far smaller than the intervals between the grains of sand, has opportunity to attach itself to one of the grains. Therefore, the finer and more numerous the particles of dirt are, the finer must be the sand and the slower must be the rate of filtration. If this rate is too great, the suspended particles flow simply through between the grains of sand. But if the sand is too fine, the filter bed may easily become water tight, but if the sand is too coarse slower filtration is to some extent a remedy. The best size of the sand grains is from one-half to one millimeter, and the sand is the better the more uniform the grains are. A sand containing much finer grains cannot be used, as it is easily rendered too compact by the pressure of the water. *Wagner's Chem. Tech.*, p. 236.

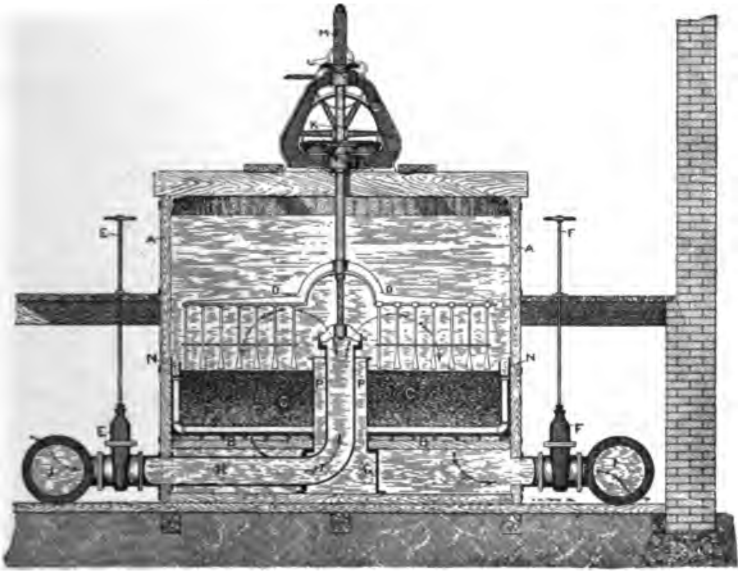

Fig. 15.

A very complete description of the sand filter beds, constructed for the Massachusetts Water Works, will be found in *The Engineering Record*, 1895. These works represent the latest advancement in this line of engineering.

A very complete article on "Purification of Sewage and of Water by Filtration," by H, F. Mills, C.E., will be found in the *Transactions of the American Society of Civil Engineers*, 1894.

To show the methods in use for quick filtration as well as the general arrangement of the apparatus, the Warren filter is taken as an example. The filter plant usually consists of a settling basin, one or more filters, and a weir for controlling the head, together with the necessary pipe connections. Each filter contains (see Figs. 15 and 16) a bed of fine sand, *C*, two feet in depth, supported by perforated copper bottom, *B*, and for cleaning this bed an agitator, *D*, is provided. This consists of a heavy rake containing thirteen teeth twenty-

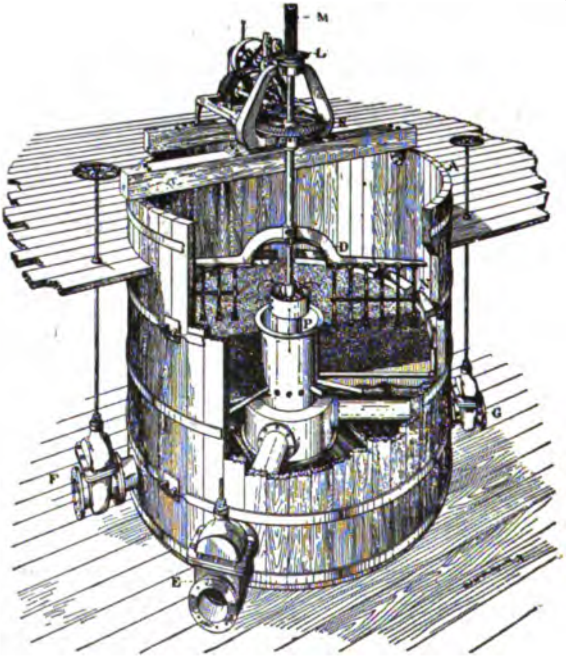

Fig. 16.

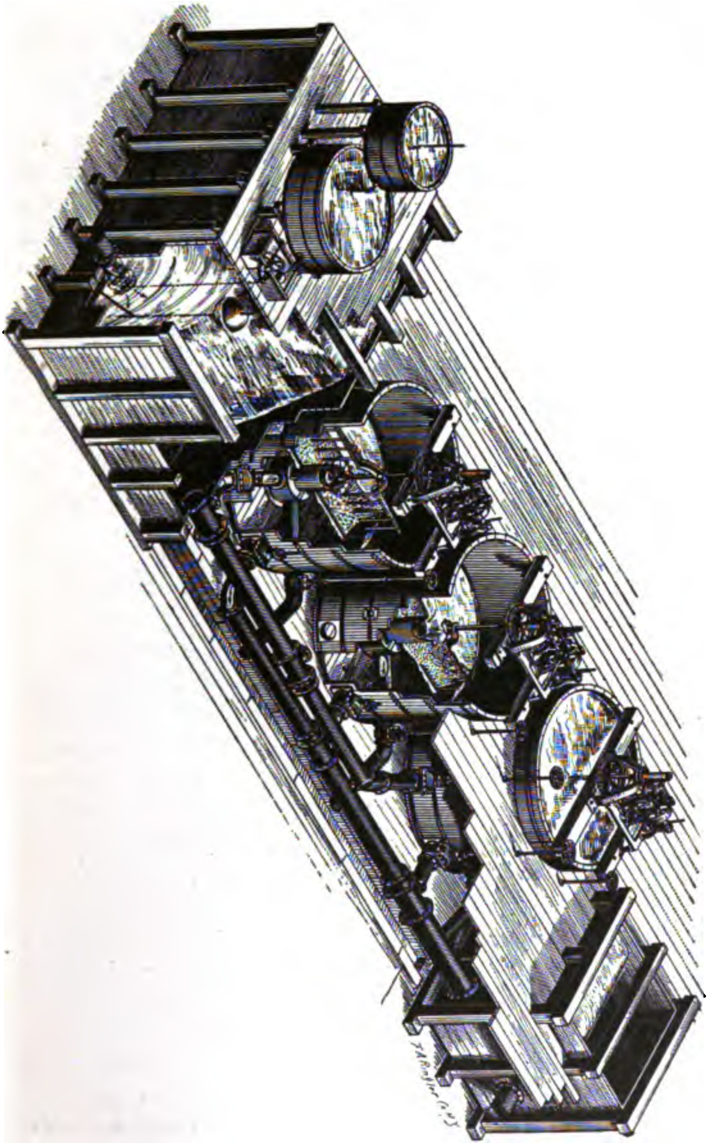

Fig. 17.

five inches long, rotated by a system of gearing, *K*, and capable of being driven into the bed by means of suitable screw mechanism, *L*, *M*, whereby the entire bed is thoroughly scoured. The process of filtration is as follows: The water enters the settling basin through a valve operated by a float, by which a constant level is maintained in the entire filter system. The water entering through this valve passes through an eight-bladed propeller of brass, from ten to sixteen inches in diameter, so arranged as to revolve freely with the passage of the water. This, by means of two small bevel gears and an upright shaft, operates an alum pump of unique design, consisting of six hollow arms radiating from a chambered hub, and bent in the direction of rotation. This pump revolves in a small tank containing a dilute solution of aluminum sulphate, or other coagulant, and by its revolution each arm takes up its modicum of alum water, passes it into the hub and to the deflector, which sends it down to the incoming water.

The latter, having received its proper amount of coagulant, is then allowed to remain in the settling basin from thirty to forty minutes, to enable the chemical reaction between the coagulant and the bases and organic matter in the water to take place, and to permit of the heavier sediment, together with a portion of the coagulated matter, to settle by subsidence to the bottom of the tank, where it can be drawn off at intervals into the sewer. The water, with all the suspended matter, as well as practically all the bacteria present in the water, bound and held together by the insoluble hydrate of alumina resulting from the addition of the coagulant, passes on through suitable piping and valves to the filter *A*, and, filling the tank, passes down through the fine zinc sand bed, leaving all the coagulated matter upon it, and makes its exit from the filter through the main *J*, bright and clear and perfectly adapted in every way for domestic purposes.

The main, collecting the filtered water from the various filters, passes along between them to the head box, or weir, over which the water is compelled to pass and which controls the operation of the filters. The top of this weir is twenty inches below the water level maintained in the filter system, and this head of twenty inches (equivalent to a pressure of three-quarters of a pound to a square inch,) is the extreme pressure that can be brought to bear upon the filters, and it is evident that they can at no time be pushed beyond the rate which experience has shown to yield the best results.

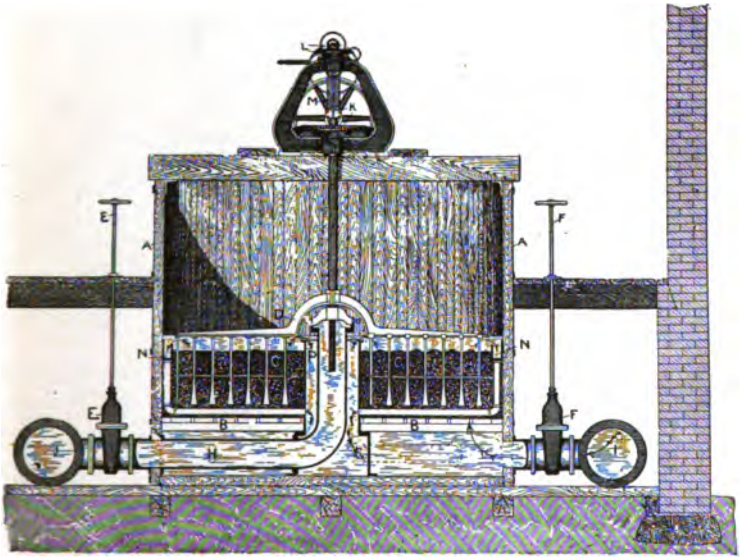

FIG. 18.

When the bed of a filter becomes clogged, and it seems best to clean it, the inlet and outlet valves *EF*, are closed, and the washout *G*, opened, allowing the contents of the tanks to escape to the sewer, Fig. 16. The agitator, *D*, is then set in motion by means of the friction clutch with which it is equipped, and as the teeth on the rake begin to plough up the surface of the bed a slight amount of filtered water is allowed to flow back up through the bed, in order to rinse off the dirt loosened by the rake. This is kept up until the rake penetrates to the bottom of the bed, and thoroughly agitates every particle of material therein.

As soon as the water following to the sewer is clear, the motion of the rake is reversed and it is slowly withdrawn from the bed. When the teeth are raised above the bed, the water pipe is closed the inlet valve *E* opened, and the filter tank allowed to fill.

After waiting a few minutes the outlet valve, *F*, is slowly opened and filtration is resumed. A filter ten feet six inches in diameter, net area, eighty-four square feet, will filter 375000. gallons of water per twenty-four hours.

Bacteriological Examination.

The bacteriological examination of water is dependent more upon the Microscopic than the Engineering Chemist.

The following references, however, are inserted:

"Micro-organisms in water" by Percy and G. C. Frankland, 1894.

"Manual of Bacteriology," by Dr. George M. Sternberg, 1892.

"A Bacterial Study of Drinking Water," by Dr. V. C. Vaughn, 1892.

"Bacteriological Diagnosis," by Dr. James Eisenberg, Vienna, 1887.

"Report of the Massachusetts State Board of Health for 1892.

"Bacteria and other organisms in water" by John W. Hill, *Transac. Amer. Soc. Civil Engineers*, Vol. xxxiii pp. 423–467.

"Practical Bacteriology" by Dr. W. Migula, London, 1893.

The Composition of Boiler Scale.[1]

The results of an analysis of boiler scale usually represent the lime and magnesia as carbonates with a portion of the former as sulphate—on the general principle that the scale made continues to exist in the form in which it was precipitated. In those portions of the boiler where the direct heat does not come in contact with it, the scale remains unchanged after formation, but the conditions are altered where the scale is subjected to intense heat. In the latter case, while the deposition of the scale-forming material at first occurs as carbonate and sulphate, the gradual heating expels some of the carbonic acid, and the oxides of calcium and magnesium are formed.

That portion of the scale nearest the iron and to the heat loses more of its carbonic acid, and becomes caustic so long as the fire continues.

As soon, however, as the fires are drawn, the oxides of calcium and magnesium become hydrated by absorption of water.

If now a sample of the scale were taken for analysis, the water of hydration becomes an important factor in the analysis.

A sample of scale from some boilers at Birmingham, Ala., gave the following result:

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[1] The scheme for analysis of Limestone, (XI), can be used in this analysis. Consult *J. Anal. Chem.* iv., Jan., 1890.

Silica and clay	11.70 per cent.
Fe_2O_3, Al_2O_3	2.81 " "
CaO	13.62 " "
MgO	41.32 " "
CO_2	6.92 " "
SO_3	0.96 " "
H_2O (of hydration)	21.78 " "
H_2O (moisture at 212° F.)	0.69 " "
Undetermined	0.20 " "
Total,	100.00 " "

An examination of this analysis shows an unusually small amount of carbonic and sulphuric acids, a large amount of water and of magnesia.

The great excess of the latter over the lime indicates that the water from which the scale was formed is a magnesia water, but its presence in this amount does not in any way alter the conditions of the problem.

With less than one per cent. of sulphuric acid and less than seven per cent. of carbonic acid, the oxides of calcium and magnesium could not exist in their entirety as carbonates or sulphates, for, combining the above acids to form carbonates and sulphates the result indicated over twenty per cent. lacking in the 100 parts.

The large percentage of the oxides of calcium and magnesium left after combination with the acids suggested water of hydration.

A sample of the scale (dried at 100° C.) was transferred to a platinum crucible and heated over the blast lamp to a constant weight. The loss of weight was over twenty-eight per cent. and, of course, included the carbonic, but not the sulphuric acid.

To check this result, a sample of the dried scale was ignited in a combustion tube and the water collected in a weighed calcium chloride tube. The result was 21.78 per cent. of water of hydration.

This satisfied the conditions existing, and the combinations gave as follows:

Silica and clay	11.70	per	cent.
Fe_2O_3, Al_2O_3	2.81	"	"
$CaSO_4$	1.69	"	"
$CaCO_3$	5.45	"	"
$MgCO_3$	7.36	"	"
$Ca(OH)_2$	13.70	"	"
$Mg(OH)_2$	56.37	"	"
H_2O (Moisture at 212° F.)	0.69	"	"
Undetermined	0.20	"	"
Total,	99.97	"	"

A section of the scale was subjected to examination, layer by layer, and the following results confirm the above.

That portion of the scale next the iron and nearest the fire contained but traces of carbon dioxide, and was principally the hydrated oxides. The middle portion of the scale was a mixture of carbon dioxide and the hydrated oxides, while the upper portion of the scale contained carbonates, but no hydrated oxides. In other words, the composition of the scale will depend, in a great measure, upon what portion of the boiler the deposit is made. That deposited on the iron or shell not in contact with the flame or not subjected to extreme heat, will remain as deposited—as carbonates and sulphates, while the scale deposited upon the iron subject to the flame or heat sufficient to drive out any carbonic acid from the scale, will vary in the amounts of carbon dioxide and water of hydration as indicated.

Scale formed in which the lime all exists as calcium sulphate and in which no magnesium carbonate is present will be subject to but little variation.

When oil has been indicated, by qualitative analysis, as present, the method of analysis requires the following modification:

The sample of pulverized scale is dried at 98° C. to constant weight, and a portion of this, one and one-half gram, is transferred, to a Soxhlet tube and the oil extracted with ether. The ether solution evaporated carefully in a platinum capsule and the amount of oil determined.

The residue in the Soxhlet tube is dried again and the analysis made in the regular way.

The following is an analysis of a boiler scale containing some lubricating oil:

SiO_2	7.36	per cent.
$Al_2O_3.Fe_2O_3$	1.91	" "
$CaCO_3$	62.71	" "
$MgCO_3$	18.15	" "
$Mg(OH)_2$	4.21	" "
H_2O. at 110°C	2.51	" "
Oil (lubricating)	3.53	" "
Undetermined	0.62	" "
Total,	100.00	" "

Nearly all waters contain foreign substances in greater or less degree, and though this may be a small amount in each gallon, it becomes of importance where large quantities are evaporated.[1]

For instance, a 100 H.P. boiler evaporates 30,000 lbs. of water in ten hours or 390 tons per month : in the comparatively pure Croton water there would be 88 lbs. of solid matter in that quantity, and in many kinds of spring water as much as 2000 lbs.

The nature and hardness of the scale formed of this matter will depend upon the kind of substances held in solution and suspension. Analysis of a great variety of incrustations show that calcium carbonate and sulphate form the larger part of all scale, that from carbonate being soft and granular, and that from sulphate hard and crystalline. Organic substances, in connection with calcium carbonate will also make a hard and troublesome scale.

The presence of scale or sediment in a boiler results in loss of fuel, burning and cracking of the boiler, predisposes to explosion and leads to extensive repairs. It is estimated that the presence of one-sixteenth inch of scale causes a loss of thirteen per cent. of fuel, one-fourth inch thirty-eight per cent., and one-half inch sixty per cent.

The Railway Master Mechanics' Association of the U. S., estimates that the loss of fuel, extra repairs, etc., due to incrustation, amount to an average of $750 per annum for every locomotive in the Middle and Western States, and it must be nearly the same for the same power in stationary boilers. When boilers are coated with a hard scale difficult to remove, it will be found that the addition of one-fourth lb. of sodium hydroxide per horse power and steaming for some hours, just before clean-

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[1] G. H. Babcock, "Steam," p. 63.

ing, will greatly facilitate that operation often rendering the scale soft and loose.

Water for Locomotive Use.

After many years of experiment upon waters for Locomotive use, by the chemists of the Chicago, Milwaukee & St. Paul R. R., the results obtained may be stated as follows:

Varieties of water may be classified by either of two methods :

1. By their chemical composition.
2. By their effect in use.

The second is manifestly what is wanted by master mechanics and superintendents.

The following may be placed in the first class:

a. Alkaline waters.
b. Non-alkaline, bad and good.

In the second class (2):

a. Those causing foaming and corrosion, but non-incrusting.
b. Hard, or incrusting.
c. Soft non-alkaline and good.

These two classes are related as follows:

"*a*" of class 1, "alkaline" waters, will produce the trouble mentioned in "*a*" of class 2; that is, foaming and in certain cases corrosion.

"*b*," the bad "non-alkaline," would be classed as hard or incrusting.

"*c*," "soft waters," would include all those having little mineral impurities of any kind.

It is, however, impossible to set hard and fast limits for each class, one generally shading into the other, and what would be called good water in the West, for instance, would be thought poor enough in the East.

In making an analysis all ingredients are grouped broadly under two heads, "incrusting" and "non-incrusting." Under the former are put such salts as are thrown out of solution by heat, and in the latter case those which do not precipitate until great concentration occurs—a condition which hardly ever happens with locomotives.

In the "non-incrusting" group is found a variety of actions. A well known property of alkali in water is to cause foaming and priming, when sudden reduction of pressure occurs upon opening the throttle. At just what point this action begins to be apparent depends on a number of circumstances. With a boiler overworked and foul from mud, it appears sooner than in one having ample heating surface, with moderate train load, uniform resistance and consequent regular consumption of steam. For a maximum allowable with good results in service and in the West, where really good water, as before mentioned, is uncommon, fifty grains per gallon of alkaline water are taken. When this figure is exceeded it certainly pays to institute a regular search for better water. With these non-incrusting salts are associated a few that are readily decomposed in contact with iron, and attack it, causing gradual corrosion. These are most commonly the magnesium chlorides and sulphates, a very small amount of which, say ten grains per gallon, should condemn the water. Organic matter is supposed also to have this action, but in the presence of alkali the danger is not great and with frequent blowing out little attention need be given it. The water may be classified as follows:

1 to 10 grains of solids per gallon, soft water
10 to 20 " " " " " moderately hard water
Above 25 " " " " " very hard water.

On this railroad "boiler compounds" are employed. Waters having thirty-five to forty grains of incrustating matter per gallon can be dealt with successfully, *provided no alkali be present.* The above reservation is made because the "compound" is itself an alkali; so in adding it to a water care must be taken not to bring the *total alkali* above, say, fifty grains per gallon, or there will be trouble from foaming. In the "Report of Analysis" blanks, directions are given fixing the amount of compound to use in each case.[1] A few examples of the different kinds of water used on this road are here given, illustrating the distinctions above drawn. The best is surface water, in the forest

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[1] This compound is a mixture of one pound of caustic soda and one-half pound of sodium carbonate, dissolved in one gallon of water. The average cost for a run of 1,000 miles being about forty cents.

region of Wisconsin; for example that from Wausau, as follows:

Total solid residue		6.78	grains per gallon.
Incrusting matter	Oxide of iron	0.23	" " "
	$CaCO_3$	2.26	" " "
	$CaSO_4$	0.56	" " "
Total		2.95	" " "
Non-incrusting matter	Organic and volatile	3.15	" " "
	Alkaline chlorides	0.68	" " "
Total		3.83	" " "

For boiler purposes this water could not be better, the incrusting matter, about three grains, being inappreciable.

For a good example of badly incrusting water, but non-alkaline, the following from Lennox Creek, Dakota, may be given:

Total solid residue		109.20	grains per gallon.
Incrusting matter	$CaCO_3$	40.31	" " "
	$MgCO_3$	7.17	" " "
Total		47.48	" " "
Non-incrusting matter	Organic and volatile	14.34	" " "
	$MgSO_4$	46.07	" " "
	Alkaline chlorides	1.31	" " "
Total		61.72	" " "

This water could not be properly purified by the addition of caustic or carbonated alkali without introducing an inadmissible amount of the latter, as above noted.

It will be noticed that the magnesium sulphate is classed as "non-incrusting" matter. It is, however, much more hurtful than the lime salts on account of its corrosive properties. The organic matter is also high, but not more so than is usual for a surface water in that locality.

For examples of absolutely worthless water, notice first, that from an artesian well at Kimball, D. T.

Total solid residue		182.06	grains per gallon.
Incrusting matter.	Calcium carbonate	61.85	" " "
	Calcium sulphate	41.44	" " "
Total		103.29	" " "
Non-incrusting matter	Alkaline sulphates	64.83	" " "
	Alkaline chlorides	13.94	" " "
Total		78.77	" " "

And again, from a 130 feet driven well at Fargo, D. T.

Total solid residue		416.84	grains	per	gallon.
Incrusting matter..	Oxides	5.00	"	"	"
	Calcium carbonate	180.00	"	"	"
	Calcium sulphate	35.46	"	"	"
Total		220.46	"	"	"
Non-incrusting matter	Magnesium sulphate	20.90	"	"	"
	Alkaline sulphates..	150.92	"	"	"
	Alkaline chlorides..	1.14	"	"	"
	Organic and volatile	23.42	"	"	"
Total		196.38	"	"	"

It is manifestly useless to attempt the purification of these waters practically.

All the round-houses are provided with hydrants and high pressure steam connections for the purpose of obtaining a powerful stream of hot water for wash-out use.

On eastern divisions, locomotives having run from 1,500 to 2,000 miles are blown off at low pressure, cooled, and the stream of hot water thrown in at hand holes, front tube-sheet and back head, and scraper worked in and out. The sediment is found mostly loose and in the form of fine mud, to the amount of ten to fifteen buckets full. After thorough cleaning, the boiler is again filled with hot water, and is ready for service.

On the western divisions the frequency of washing out is increased, doing so as often as once every 300 or 400 miles run. As to the economy of using hot water *always*, there can be no question. Fully seventy-five per cent. in the number of cracked fire-box sheets are saved by this practice alone, and it materially reduces the force of repairers in round-houses, notwithstanding a very large increase of engine mileage.

Many people are opposed to the use of chemicals in boilers, rightly upon general principles; but when the proper ones are used, the experiments have failed to show the slightest injury therefrom, while the economy resulting, both in service and repairs, has amounted to an enormous sum on this system.

Feed Water Heaters.

Feed water heaters as well as boiler economizers are often used as eliminators of the scale-forming materials in water. This is due to the fact that waters containing much calcium and magnesium carbonates when heated to the usual temperature in feed water heaters (200°–210°F), give up the excess of carbon dioxide that holds the calcium and magnesium carbonates in solution, and the latter are precipitated and removed before the water enters the boiler.

Where calcium sulphate is a large constituent of the water, feed water heaters using exhaust steam do not precipitate the lime salt, but if the feed water be heated by live steam under pressure to a temperature of 240° F, then the calcium sulphate precipitates. The addition to the water of the necessary amount of sodium carbonate will precipitate the lime as carbonate, at ordinary temperatures, and it will thus be found more economical in this case to use feed waters heaters, using exhaust steam with sodium carbonate, than feed water heaters using live steam only.

An example of an upright feed water heater heated by exhaust steam is the "Goubert."

Fig. 19.
The Goubert feed-water heater.

Fig 20.
(Vertical type.)

The exhaust steam from the engine is admitted to the shell through the nozzle on one side and spreading between the brass tubes, impinges upon them on its passage across to the outlet on the opposite side: the water of condensation being removed by the drain pipe. The cold water may be admitted at either top or bottom of the heater, passing out at the opposite end: but for bad waters the feeding should be at the top. This being a closed heater and the water being forced through against boiler pressure, the flow along the heating tubes will be the same whether the water moves in an upward or a downward direction, but in the latter case the separation and settling of sediment will be much more thorough, while the heating will be the same.

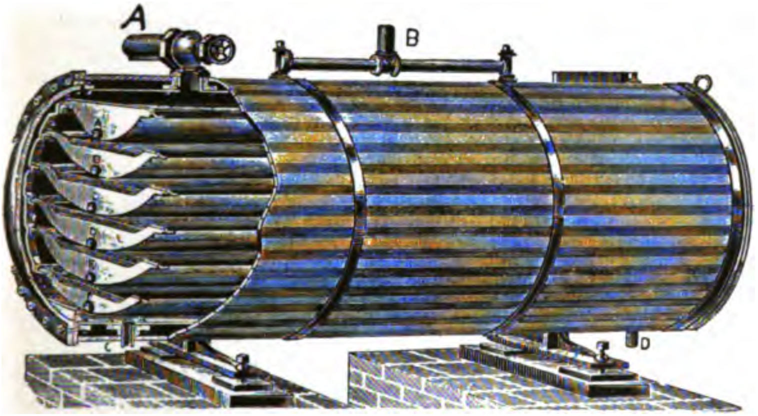

Fig. 21.—The Hoppes feed-water purifier.

This purifier consists of a round shell of best boiler steel, having a solid pressed flange steel head riveted in the back end, and a solid pressed flange steel head bolted to a heavy ring on the front end, by studs and nuts. Within the shell are a number of trough-shaped pans or trays, placed one above another, and supported on steel angle ways, fixed longitudinally by means of brackets to the sides of the shell. These pans are formed from thin sheet metal, the heads or end pieces being malleable iron, whereby a very light,strong and durable construction is obtained, and a degree of elasticity secured to the pans, which permits the lime or other incrustations being easily removed. Six pans are placed in a tier, and from one to four tiers used, according to capacity required. The purifier is connected to the boiler by a large steam pipe *A*, and the exit pipe *D*. A blow-off pipe is also connected at *C*. The feed pipe from the pump or boiler feed is attached at *B*.

In operating the purifier, the water is pumped in at *B* and distributed into the upper pans through the pipes leading into each pan. While the purifier is in operation, the pans remain full of water, and afford ample settling chambers for the heavier solids, such as mud, sand etc., etç., while the carbonates and sulphates (scale-forming) adhere to their under sides.

An analysis of a sample of water before passing through one of these heaters at Rochester, N. Y., is as follows:

BEFORE USE.

Inorganic solids	128.74	grains per gallon.
Organic matter	3.38	" " "
Total solids	132.12	" " "

AFTER PASSING THROUGH HEATER.

Inorganic solids	8.44	grains per gallon.
Organic matter	3.20	" " "
Total solids	11.64	" " "

TABLE SHOWING THE YEARLY SAVING EFFECTED BY THE USE OF THE FEED-WATER HEATER FOR VARIOUS HORSE POWERS AND AT DIFFERENT PRICES OF COAL.

Horse power of engine.	Coal consumption at 4 pounds per H. P. per hour.		Saving of 13½ per cent.	Price of coal per ton of 2240 pounds.									
	Daily.	Yearly.		$1.50	$2.00	$2.50	$3.00	$3.50	$4.00	$4.50	$5.00	$5.50	$6.00
	lbs.	tons.	tons.										
50	2000	268	36.18	$ 54	$ 72	$ 90	$ 108	$ 126	$ 145	$ 163	$ 181	$ 199	$ 217
60	2400	321	43.33	65	87	108	128	152	173	194	217	238	260
70	2800	375	50.62	76	101	126	152	177	202	227	253	278	304
80	3200	429	57.91	87	116	145	174	203	232	261	289	318	347
100	4000	536	72.36	108	145	187	217	253	289	325	362	398	434
120	4800	643	86.80	130	174	217	260	304	347	390	434	477	521
160	6400	857	115.69	173	231	289	347	404	463	520	578	635	694
200	8000	1072	144.72	217	289	362	434	506	579	651	724	796	868
250	10000	1340	185.90	279	372	465	558	651	744	837	929	1022	1115
300	12000	1608	226.08	339	452	565	678	791	904	1017	1130	1243	1356
350	14000	1876	253.26	380	506	633	760	886	1013	1139	1266	1392	1519
400	16000	2144	289.44	434	579	723	868	1013	1158	1302	1447	1591	1730
500	20000	2680	361.80	543	724	904	1085	1267	1447	1627	1809	1990	2170
600	24000	3216	433.30	650	867	1083	1300	1517	1733	1950	2170	2387	2600
700	28000	3752	506.20	759	1012	1265	1518	1771	2025	2278	2531	2784	3037
800	32000	4288	579.10	868	1158	1448	1737	2026	2316	2605	2895	3184	3474
900	36000	4824	651.24	977	1302	1628	1954	2279	2605	2930	3256	3581	3907
1000	40000	5360	723.60	1085	1447	1809	2170	2532	2894	3255	3618	3990	4341

PERCENTAGE OF FUEL SAVED BY HEATING FEED WATER.

(Steam pressure 60 pounds.)

Initial temperature of water entering heater.	Heat units per lb. absorbed in generating steam.	Temperature of water entering boiler.												
		120° F.	140° F.	160° F.	180° F.	200° F.	202° F.	204° F.	206° F.	208° F.	210° F.	212° F.	214° F.	216° F.
32°	1175	7.49	9.19	10.89	12.59	14.30	14.47	14.64	14.81	14.98	15.15	15.32	15.49	15.66
40°	1167	6.86	8 57	10.28	12.00	13.71	13.88	14.05	14.22	14.40	14.57	14.74	14.91	15.08
50°	1157	6.05	7.78	9.51	11.24	12.97	13.14	13.32	13.49	13.66	13.83	14.00	14.18	14.35
60°	1147	5.23	6.97	8.72	10.46	12.21	12.38	12.55	12.73	12.90	13.08	13.25	13.43	13.60
70°	1137	4.41	6.16	7.91	9.67	11.43	11.61	11.78	11.96	12.14	12.31	12.49	12.66	12.84
80°	1127	3.44	5.32	7.10	8.87	10.65	10.82	11.00	11.18	11.36	11.53	11.71	11.89	12.07
90°	1117	2.68	4.47	6.26	8.06	9.85	10.03	10.21	10.38	10.56	10.74	10.92	11.10	11.28
100°	1107	1.80	3.61	5.42	7.23	9.03	9.21	9.39	9.57	9.75	9.93	10.11	10.29	10.47
110°	1097	0.91	2.73	4.55	6.38	8.20	8.38	8.58	8.74	8.93	9.11	9.29	9.47	9.66
120°	1087	....	1.84	3.67	5.51	7.35	7.54	7.77	7.90	8.09	8.27	8.45	8.64	8.82

"Blowing-off." The arrangement in a boiler for this purpose usually consists of one or two internal pipes extending along the bottom of boiler and connected with the blow-out tap. They are placed about one and one-half inches clear of the plates and are perforated on their under side. It is usual to blow-out the sediment every two or three days just before drawing the fires and the sediment in the water has had time to settle. Consult also "Wilson on Boilers," page 169–171.

Use of Chemicals and Filtration.

Dervaux Water Purifier for boiler use.

This apparatus (Figs. 22, 23) is automatic in action, and is thus described.[1] The purifier is intended to act as an eliminator for both calcium sulphate and calcium and magnesium carbonates. It not only acts to precipitate the dissolved impurities, but also to collect those that are in suspension. These last are caught and held in the tower-shaped holder *D*. The water enters at *H*, passes down through *E*, and is made to rise through a series of funnels or inclined funnel-shaped walls.[2] On these walls the coarsest particles are caught and from them they flow down to the bottom of the tower, where they collect: the water then passes upwards though the filters *F*, which are made of wood shavings, and flows off, freed from its mechanical impurities through the opening *T*. In the mean time, by the addition of lime and soda, the water has been chemically purified in the following way: The water first flows into the reservoir *C*, through the pipe *H*. In *C*, there is a float for regulating the flow of water. A portion of the water goes into *E*, through the pipe *P*, while the rest passes through the valve *V* into the lime saturator *S*; *S* is filled with lime: the water first meets the lime at the bottom of the saturator and passes up through it; the conical shape of *S* causes the rise to be slower and slower as the water nears the top, so that the milk of lime, at first formed, has plenty of time to clarify itself. The lime water usually contains some calcium carbonate in suspension: and as this is worthless for purposes of purification, it is eliminated by causing the water to flow over

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[1] *Papier Zeitung*, 34, 984.

[2] The Chemistry of Paper Making, p. 345.

Fig. 22.

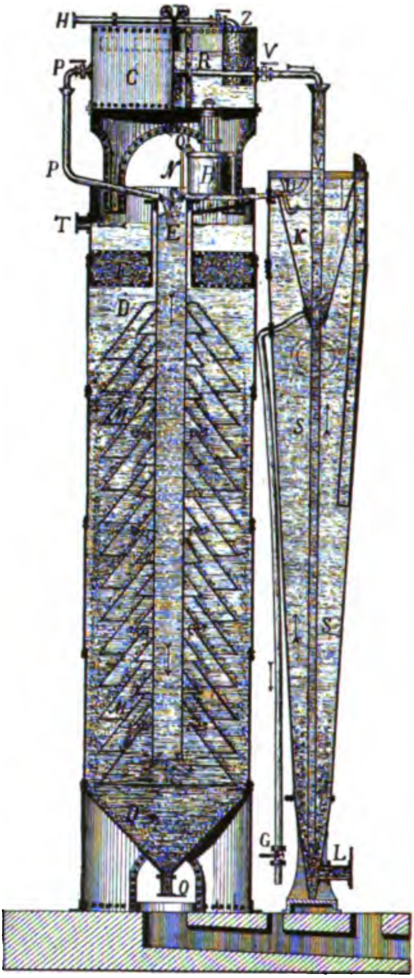

Fig. 23.

into the cone *K*, which is closed at the bottom. In this cone the carbonate settles out, and may be drawn off through *G*. The clear, saturated lime-water, containing 1.3 gram of lime per liter, runs then directly into the mixing tube *E*. A solution of soda-ash is made by taking a known weight of the ash, which is placed in the tank *Z*, after which the tank *K*, is filled to a definite mark with water. This solution slowly passes through the tube provided with strainers: a float in the tube keeps the water in *E* at a constant level. The siphon *N*, one end of which dips to the bottom of *B*, allows the alkaline solution to flow into *B*. The regulation of the flow in *E* is performed as follows: The siphon *N* is joined by a chain *Q*, to the float in *C*. In case the flow of water through *H* to *C* is cut off, the float sinks, raising *N* and thus stopping the flow of the solution. At the same time the level in *C* sinks so low that the flow of water through *P* and *V* ceases: as soon as the flow of water through *H* recommences, the apparatus is again set in operation automatically.

The chemical operations may be stated as follows: The addition of the lime softens the water by precipitating any bicarbonate which may be present, and the excess of lime is thrown down by the sodium carbonate. This, by its precipitation throws out much of the finely divided organic impurity. The apparatus may be easily modified to work with alum where desirable.

This Derveax Purifier is extensively used in France and Germany.

In England the apparatus devised and patented by L. Archbutt, F. I. C., and R. M. Deeley, M. E., has an extensive use for the purification of boiler waters.

The drawings (Figs. 24, 25, 26) show the construction and represent a purifier suitable for the treatment of from 5,000 to 10,000 gallons of water per hour. It consists of a cast-iron tank, measuring 32 feet × 16 feet × 10 feet deep, divided into two equal parts by a transverse partition of cast or wrought iron. The two tanks are intended to be used alternately, so as to maintain a continuous supply of softened water.

The water to be purified is admitted to either tank by means of the supply pipe, 1, which is connected up to a pump or main. The water fills up nearly to the level of the top of the well, 4. While the tank is filling the proper amounts of lime and sodium carbonate are weighed out, with the addition, in some cases, of a very small quantity of aluminum sulphate, or alumina-ferric cake, and these are boiled up with water in the small chemical tank, 2, by means of steam from the steam pipe. The trajector, 3, is put into action by opening its steam valve, and then the

PATENT HARD WATER PURIFIER.

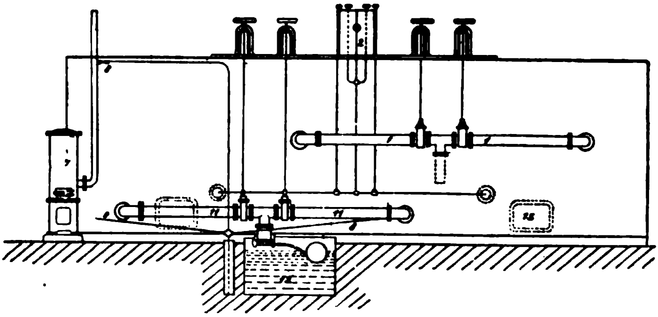

Fig. 24.

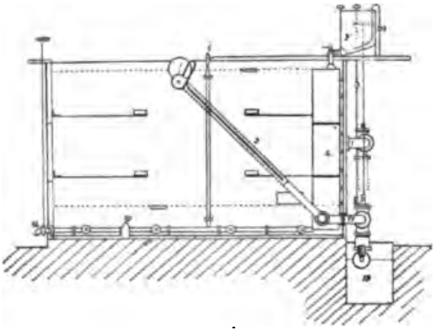

Fig. 25.

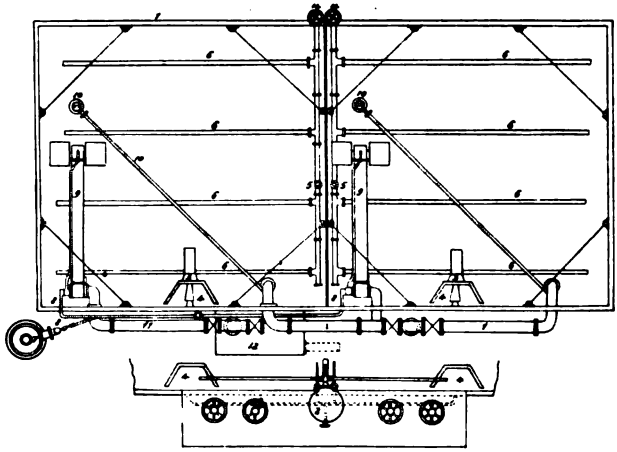

Fig. 26.

chemical liquid is run out of the chemical tank into the well. The trajector creates a powerful current of water from the well, through the projecting pipe, across the tank, and into this current the chemicals pass. After the chemicals have thus been added and mixed with the water, and the trajector shut off, steam is admitted to the blower, 5, which causes air to be sucked down the orifice and forced out of the perforations in the pipes laid close to the bottom of the tank. After the blower has been in operation for fifteen minutes, the steam is turned off and the water is allowed to rest. The result is that in about thirty minutes very nearly all of the precipitate will have settled to the bottom of the tank. The drawing-off and carbonating are operations that are automatically and simultaneously effected by means of the floating discharge pipe, 9, of rectangular section.

Fuel gas, from the coke stove, 7, constructed so as to produce a minimum of carbon monoxide and a maximum of carbon dioxide is forced continuously by means of a very small steam blower, 8. The gas and water pass together through the ball tap fixed over the small supply tank, 12, into which the softened and carbonated water falls, and from which it is drawn off for use, whilst the residual gas and nitrogen, etc., escape into the air. The mud is removed by extending the main blower pipe through the side of the tank where it terminates in a valve, 14, which by opening for a few minutes at intervals the accumulation of mud is prevented.

The reasons for carbonating the softened water are fully explained in a paper read before the Society of Chemical Industry in June, 1891. Uncarbonated softened water often forms a deposit in pipes and especially in the feed apparatus of steam boilers, which may become very troublesome. This is not a peculiarity of water softened in this apparatus.

The output can be calculated as follows:

$v =$ the number of gallons of softened water supplied continuously per hour.

$x =$ the working capacity, in gallons, of each tank.

$y =$ the number of minutes required to fill each tank.

$z =$ the number of minutes required for settling.

$$v = \frac{60\,x}{25 + y + z} = \text{the cost.}$$

Grains per Gallon.	1	2	3	4	5	6	7	8	9
Calcium carbonate	8.74	13.15	16.59	10.89	9.19	2.06	9.41	8.34	1.39
Magnesium carbonate	2.78	0.33	0.31	2.76	1.40	0.94	1.00	2.82	1.78
Calcium sulphate	3.26	....	4.30	2.99	12.17	47.34	22.91	40.61	54.14
Magnesium sulphate	....	1.96	1.28	12.41	7.05	5.70	15.90	22.25	22.46
Sodium sulphate	1.44	0.30	....	18.96	....	9.98	....	2.65	28.96
Magnesium nitrate	....	....	....	....	13.69	....	11.50	....	....
Sodium nitrate	....	0.96	small	....	....	...	....	small	....
Magnesium chloride	....	....	....	....	0.64	....	2.08	....	....
Sodium chloride	2.72	2.06	3.05	5.28	6.30	6.77	5.05	6.35	5.28
Silica	0.43	0.39	0.42	0.31	0.62	0.62	0.90	0.84	0.36
	19.37	19.15	25.95	53.60	51.06	73.41	68.75	83.86	114.37
Total lime (CaO)	6.24	7.36	10.95	7.39	10.16	20.64	14.70	21.39	23.07
Total magnesia (MgO)	1.33	0.81	0.58	5.48	7.02	2.36	9.82	8.81	8.38
Total hardness (=calcium carbonate-equivalent to total lime and magnesia)	14.50	15.16	20.99	26.77	35.53	42.7	50.57	60.02	61.95
Cost of chemicals required for softening 1000 gallons	1½c.	1½c.	2c.	4c.	7c.	9½c.	10c.	12c.	14c.

NOTE.—The above estimates of cost are based upon the following prices, *viz.:*

Quicklime..................at $4.75 per ton. | 58% Soda ash..........at $34 per ton.

To remove calcium carbonate by chemical means from water costs very little, because lime alone is necessary, and is very cheap. To remove calcium sulphate, alkali must be used, which greatly increases the cost. Both lime and alkali are necessary for the removal of certain magnesium salts, and the alkali must be used in greater relative proportion. Waters containing much magnesium salts are therefore the more costly to treat. The table on page 110 gives the analyses of nine typical samples of water, together with the cost of chemicals needed to soften each by this process, and reduce the hardness to 3° and not exceeding 5°.

Filter Presses.

Filter presses are often used for rapid filtration of water. These presses consist of a number of filter chambers with solid separating walls, which are held between two head pieces, one of which is fast and the other movable; the latter as well as the filtering frames slide along two strong iron rods. Between the chambers the filtering cloth is hung and this also helps to make the outer edges fit closer together. The whole system is pressed together by a screw or lever or by hydraulic pressure and forms a number of hollow spaces lying together and communicating with one another. Between these hollow spaces the liquid to be filtered is pressed by a pump or other means. During this process the separation of the liquid and solids takes place, in that the liquid

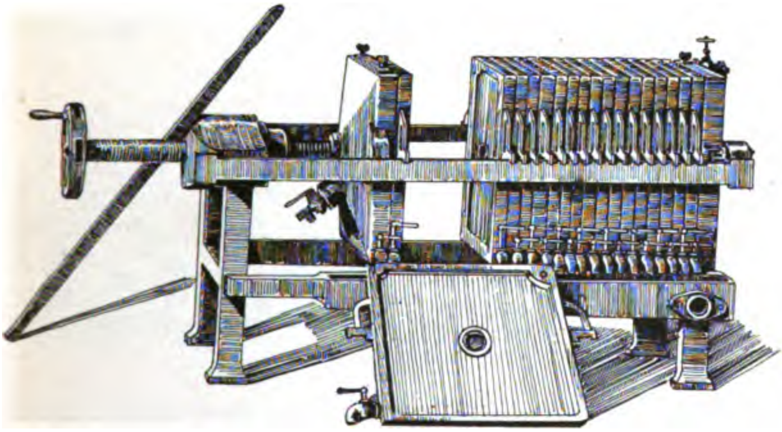

Fig. 27.

is forced through the cloth and runs out clear through channels to a common outlet, leaving the solids behind.

We distinguish two varieties of filter presses.

1. Chamber Presses, (Fig. 27) by which the space for the cake *i. e.*, the solid matter remaining, is formed by the edges of each two filters plates, so that the cake falls out when the press is opened.

2. Frame Presses, by which the space for the cake is formed by frames that are placed between each two filter plates, so that the cake can be lifted out with the frames.

In order to dry the cake completely and to wash it, when necessary, there are in most filter presses two extra canals in each chamber, one in which the washing fluid enters and the other by which it is removed. There is also an attachment by which liquids can be filtered hot or cold.

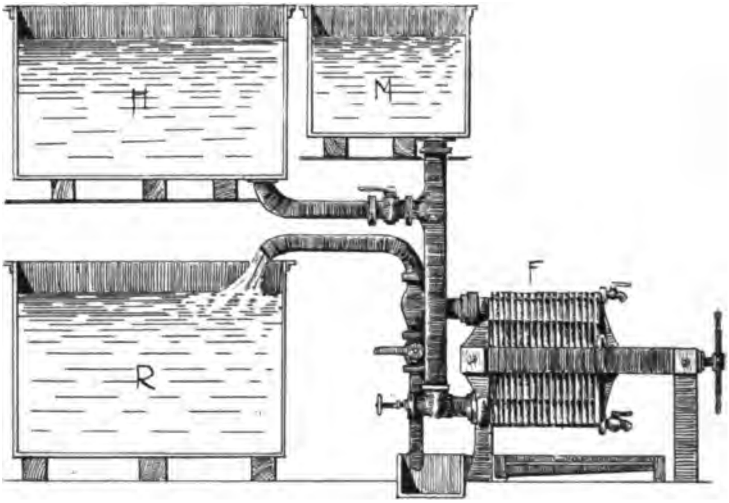

Fig. 28.

The Porter-Clarke process for softening hard water, largely used in England, makes use of filter presses to remove the precipitated material in the water. Where this latter precipitate is very fine and small in amount, manufacturing establishments sometimes arrange a system as shown in Fig 28 in which fibers of cellulose are added to collect the fine precipitate. The apparatus consists of a high horizontal reservoir H (Fig. 28) for reception of the water to be filtered, another reservoir or tank M, in which the floating material (or fibers of cellulose or asbestos) is mixed with water, a reservoir R into which the purified water flows and the filtering apparatus proper F. The latter is composed, as are the filter presses, of a series of frames on which metal sieves are fastened. The filtration takes place in the following manner: The thin mass of cellulose or asbestos fibers are caught by the sieves and remain on them; the water is then allowed to pass from the reservoir H through the sieves which now holds back all suspended matter, so that clear water flows in the reservoir R.

Another method made use of in some large industrial plants, is to combine the action of a heater, chemical precipitation and filtration by filter-presses as shown in Fig. 29.

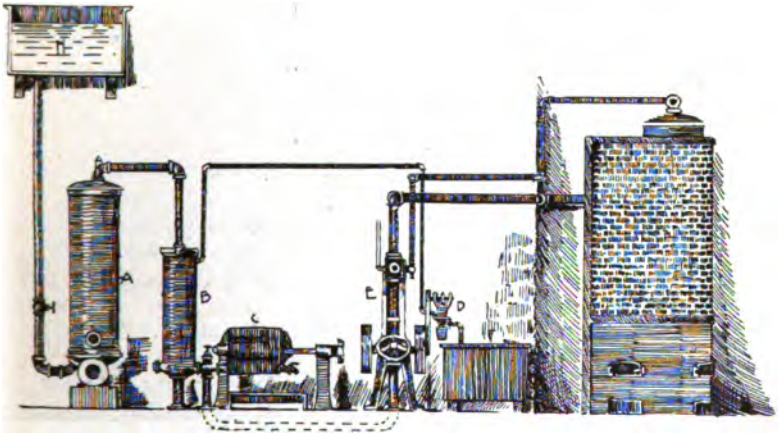

Fig. 29.

The water passes first through the heater A in which it is brought to the temperature of the heater, thence into the precipitation tank B in which it is mixed with the chemicals in solutions the latter being pumped into B from F by means of the pump D. The water then passes into the filter press C, in the chambers of which the suspended matter is retained, and is then pumped by the pump E either directly to the boiler or else to a reservoir. The water and chemicals are mixed in the proportions shown to be necessary by analysis. This system of water purification has shown itself to be very successful, but the filter press must be cleaned every two to eight days according to the composition of the water.

References: "Boiler Deposits," Vivian B. Lewes, F.C.S., *Transactions Inst. of Naval Architects.* Vol. XIV.

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"Corrosion of Steam Boilers." By David Phillip, *Proceedings Institution of Civil Engineers*, **65**, 73.

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"The Treatment of Hard Water." By L. Archbutt, F.I.C., and R. M. Deelay. *J. Soc. Chem. Industry* **10**, 511.

"The Purification of water, on the large scale, by means of Iron." By William Anderson. *Proceedings of the Institution of Civil Engineers.* **81**, 279.

XVI.

Determination of the Heating Power of Coal and Coke.

The simplest method, but which gives only approximate results, is the ignition of coal with litharge in a crucible. In detail the process is as follows: one gram of the finely powdered coal is intimately mixed with thirty grams of litharge (PbO), transferred to a No. 3 Hessian crucible, twenty grams more of litharge placed on top of the charge, the crucible covered and heated at a high heat in the furnace for fifteen minutes. The crucible is removed, allowed to cool, broken, and the button of metallic lead cleaned from the slag and carefully weighed.

Duplicate results should not vary more than 0.025 gram. To calculate the result:

One part of carbon reduces thirty-four times its weight of lead, and if one kilo. of carbon = 8140 calories, then each part of lead is equivalent to $\frac{8140}{34} = 239$ calories.

Suppose the lead button from one gram of coal weighed 31.05 gram, then $\frac{8140}{34} \times 31.05 = 7420.9$ calories per kilo. or 13357.7 B. T. U. per pound of coal, which represents the heating power of the coal.

The heating power of coke, containing no volatile combustible matter, can be calculated from the analysis, thus

Carbon	94.43	per cent.
Ash	5.57	" "
	100.00	" "

$\frac{94.43}{100} \times 8140 = 7686.6$ calories = 13837 B. T. U. per pound.

Bituminous coals contain volatile combustible matter as well as varying amounts of water, for which reasons both of the above methods give very incorrect determinations of the heating power.

Three methods are available (which include all varieties of coals:)

1. Calculation of the heating power from the results of an elementary analysis of the coal, *viz.*: determination of the percentages of carbon, hydrogen, nitrogen, oxygen, sulphur and ash.

2 The use of calorimeters in which a sample of coal is burned and its heating power determined directly from the experiment.

3. The combustion of large amounts of coal in specially designed apparatus therefor, and also boiler tests.

Calculation of the Heating Power from the Results of an Elementary Analysis of the Coal.

a. Determination of the carbon and hydrogen. Select a Bohemian glass combustion tube about seventy cm. long, two cm. in diameter, open at both ends (Fig. 30). Place in it at *j*

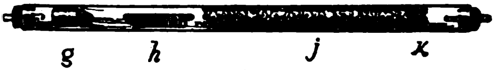

Fig. 30.

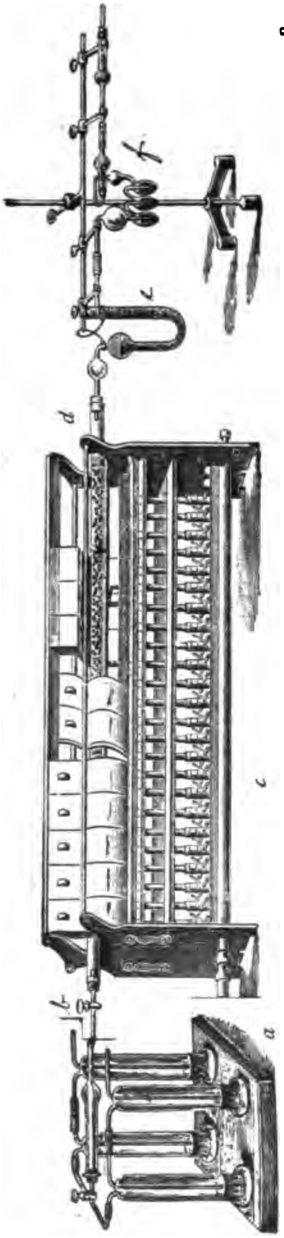

Fig. 31.

granulated cupric oxide for a distance of about thirty cm., and at *k* a plug of asbestos; place the tube in a combustion furnace *c*, connect it at *b* with the drying apparatus *a*, and at *d* with calcium chloride tube *e* filled with $CaCl_2$, granulated. The latter is connected with an aspirator, and air is very slowly drawn through the apparatus; at the same time the furnace is gradually lighted and the heat increased until all the cupric oxide has reached a red heat. Maintain this for fifteen minutes, turn off the gas, and continue the aspiration of air until the tube is nearly cold. This preliminary heating is necessary to eliminate any moisture that may be in the tube or in the cupric oxide.

Transfer five-tenths gram of the finely powdered coal to a weighed porcelain boat and place in the tube at *h*; at *g* is a coil of platinum foil. The calcium chloride tube *e* (Fig. 31) is now accurately weighed, as well as the potash bulbs *f*,[1] and when all the connections are properly made, heat is turned on in the furnace at the end *d*, and oxygen gas is very slowly passed through the apparatus. At intervals of a few minutes the heat is turned on in the furnace until the cupric oxide is at a red heat, and finally the entire tube from *k* to *g* is also at that temperature.

After the complete combustion of

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[1] The latter one-third full of KOH solution sp. gr. 1.27.

the carbon of the coal, which is indicated by the absence of black particles in the porcelain boat, turn off the heat in the furnace, but continue the slow current of oxygen until the apparatus is nearly cold. The hydrogen in the coal by its combustion is converted into water and absorbed by the calcium chloride tube *e*; the carbon of the coal, by its combustion with excess of oxygen has produced carbon dioxide, and is absorbed in the potash bulbs *f*. From the increase of weights thus obtained the percentages of hydrogen and carbon are calculated, thus:

Amount of coal taken = 0.500 gram.
Calcium chloride tube + H_2O = 36.5118 grams.
" " " = 36.4025 "
H_2O = 0.1093 "
0.109 gram H_2O = 0.0121 gram H.
$\frac{0.0121 \times 100}{0.500} = 2.42$ per cent. hydrogen.
The potash bulbs and CO_2 = 34.9554 grams.
" " " = 33.3200 "
1.6354 "
1.6354 grams CO_2 = 0.4460 gram C.
$\frac{0.4460 \times 100}{0.500} = 89.20$ per cent. carbon.

The ash is as follows:

Remove the porcelain boat from the combustion tube carefully and weigh; the increase of weight is ash. Thus:

Porcelain tube + residue (ash) = 8.9693 grams.
" " = 8.9460 "
Ash = 0.0233 "
$\frac{0.0233 \times 100}{0.500} = 4.66$ per cent. ash.

b. The nitrogen determination is made as follows:

Select a combustion tube about sixty cm. long and two and five-tenths cm. diameter, drawn to a point at one end and open at the other end (Fig. 32).

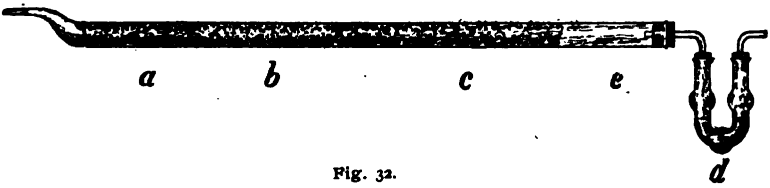

Fig. 32.

At *a* place three grams of crystallized oxalic acid, then a few layers of freshly ignited soda-lime, and at *b* insert five-tenths gram of the powdered coal mixed with about twenty grams of soda-lime, fill the rest of the tube with soda-lime and finally some asbestos near the open end of the tube. Connect with a bulb tube *d* containing fifteen cc. of a standard solution of sulphuric acid, each cc. of which contains 0.049 gram sulphuric acid.

The combustion tube is now placed in the combustion furnace and heat is gradually applied under the tube at *e* and extended slowly towards *a*. The soda-lime between *c* and the coal must be at a red heat before heat is applied under the coal. Now heat the tube until the soda-lime and the coal are well heated and maintain this until no more gas is generated or passes into the standard acid; being careful, of course, that none of the oxalic acid has yet been heated.

Gradually heat the oxalic acid, which slowly vaporizes, and in passing over the soda-lime is converted into carbon dioxide. The nitrogen in the coal, by this ignition with soda-lime, is converted into ammonia and forced out of the tube into the standard acid by the excess of carbon dioxide generated from the oxalic acid.

After the operation is completed, disconnect the U-tube containing the standard acid, transfer its contents to a No. 3 beaker, add a few drops of litmus solution and titrate with normal soda solution to determine the amount of ammonia united with sulphuric acid. Thus:

Coal taken = 0.500 gram (dried)
H_2SO_4 solution taken = 15 cc.
Normal soda solution required to neutralize free acid = 14.768 cc.

(One cc. NaOH solution neutralized one cc. H_2SO_4) 0.232 cc. of H_2SO_4 solution neutralized by the ammonia.

If one cc. H_2SO_4 solution = 0.049 gram H_2SO_4 : : 0.232 cc. = 0.0113 gram H_2SO_4.

0.0113 gram H_2SO_4 = 0.00392 gram NH_3.
= 0.00322 " N.

$$\frac{0.00322 \times 100}{0.500} = 0.65 \text{ per cent. nitrogen.}$$

The method of Kjeldahl can also be used for the determination of nitrogen in coal. Consult "Contribution a l'etude des combustibles," P. Mahler, 1893, p. 19.

The sulphur is determined as directed in scheme XII, and in this sample amounted to 0.19 per cent.

Having determined all of the constituents in the dried coal but oxygen, the latter is estimated by subtracting the sum of the other constituents from 100. Thus:

Carbon	89.21	per cent.
Hydrogen	2.43	" "
Nitrogen	0.65	" "
Sulphur	0.19	" "
Ash	4.67	" "
Oxygen	2.85	" "
Total	100.00	" "

d. We will now include in this analysis the hydroscopic water (the above analysis having been made upon the dried sample).

This moisture in the coal is a direct loss in the calorific power, since it absorbs heat.

Amount of coal taken	2 grams.
Watch-glass and coal before drying twenty minutes at 102° C	12.162 grams.
Watch-glass and coal after drying twenty minutes at 102° C	12.101 "
Loss (moisture)	0.061 "

$$\frac{0.061 \times 100}{2} = 3.05 \text{ per cent. moisture.}$$

The complete analysis of the coal will now be:

Moisture	3.05	per cent.
Carbon	86.49	" "
Hydrogen	2.36	" "
Nitrogen	0.63	" "
Sulphur	0.18	" "
Oxygen	2.76	" "
Ash	4.53	" "
Total	100.00	" "

The calorific power of the coal is calculated from the following data:

A calorie is the standard heat unit, and represents the heat required to raise the temperature of one kilo of water from 4° C. to 5° C.

A British thermal limit ("B. T. U.") is the heat required to raise the temperature of one pound of water 1° F., at its temperature of maximum density, (39.1°).[1] To reduce calories per kilo to "B. T. U." per pound, multiply by $\frac{9}{5}$.

One kilo of carbon (from wood charcoal) in burning to carbon dioxide produces 8140 calories.

These figures, 8140, obtained by Berthelot and Bunte are probably nearer correct than the figures 8080 given by Favre and Silbermann.

One kilo of sulphur in burning to sulphur dioxide produces 2220 calories.

One kilo of hydrogen in burning to water (condensed) produces 34500 calories.

If the water produced by the burning of the hydrogen is not condensed, but remains in the form of steam, part of the 34500 calories, produced by the combustion of one kilo, appears as latent heat and as sensible heat in the steam. Thus, suppose one kilo of hydrogen and eight kilos oxygen, both at 15° C. unite to form nine kilos of steam which escapes at 100° C.

The total heat of one kilo of steam at 100° C., measured from water at 15° C. is 622.1 calories, and of nine kilos, $9 \times 622.1 = 5599$ calories, which subtracted from the 34500 calories produced by the combustion of one kilo of hydrogen, leaves 28901 calories as the available heat of combustion of hydrogen at 15° C. when the product of combustion escapes as steam at 100° C.

If the steam escapes at some other temperature, or if the ini-

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[1] One French calorie =3.968 British thermal units : one B. T. U. = 0.252 calorie. The "pound calorie" is sometimes used by English writers : it is the quantity of heat required to raise the temperature of one pound of water 1°C, one pound calorie = 2.2046 B. T. U. = $\frac{9}{5}$ calories.

The heat of combustion of carbon, to CO_2, is said to be 8140 calories. This figure is used either for French calories or for pound calories as it is the number of pounds of water that can be raised 1°C. by the complete combustion of one pound of carbon, or the number of kilograms of water that can be raised 1°C. by the combustion of one kilogram of carbon. [Kent].

tial temperature of the hydrogen is other than 15° C. the available heat units will vary accordingly.

In *practical* calculations of the heating value of fuel, it is generally most convenient to take the total calorific power of the hydrogen it contains at 34500 calories per kilo, and after obtaining the total heating value of the fuel on this basis to make the necessary corrections for the initial temperature of the hydrogen and for the latent and sensible heat of the steam in the products of combustion.

The heating value of coal is thus calculated from the analysis:

Let C = the percentage of carbon in the coal.
Let H = " " " hydrogen " "
Let O = " " " oxygen " "
Let S = " " " sulphur " "

Then:

$$\text{Heating power} = \frac{8140\,C + 34500(H - \frac{O}{8}) + 2220\,S.}{100}$$

$$= \frac{(8140 \times 86.49) + 34500\,(2.36 - 0.345) + 2220 \times 0.18}{100}$$

$$= \frac{704028 + 69517.5 + 399.6}{100}$$

= 7739.4 calories per kilo of coal.

Where the products of combustion of hydrogen escape as steam at 100° C., the formula will be:

$$\text{Heating power} = \frac{8140\,C + 28901\,(H - \frac{O}{8}) + 2220\,S - 622\text{W}}{100}$$

W = moisture of the coal.

Then:

$$= \frac{8140 \times 86.49 + 28901\,(2.36 - 0.345) + 2220 \times 0.18 - 622 \times 3.05}{100}$$

$$= \frac{704028.6 + 58235.5 + 399.6 - 1897.3}{100}$$

= 7645.6 calories per kilo of coal.

To calculate the amount of air required for complete combustion, the following data are required:

1 kilo of carbon burning to carbon dioxide requires 2.66 kilos of oxygen.
1 " " hydrogen " " water " 8.00 " " "
1 " " sulphur " " sulphur dioxide " 1.00 " " "

Air is composed of a mechanical mixture of oxygen and nitrogen in the proportion by weight, of 26.8 parts of nitrogen with eight parts oxygen ; that is, 3.35 parts of nitrogen with one part of oxygen; or in volumes 3.76 cubic meters of nitrogen with one cubic meter of oxygen.

The volume of 1 kilo of oxygen is 0.74 cubic meter at 16.67°C.
" " " " nitrogen " 0.84 " " " " "
" " " " hydrogen " 11.84 " " " " "
" " " " sulphur dioxide " 0.36 " " " " "
" " " " carbon dioxide " 0.54 " " " " "
" " " " air " 0.82 " " " " "

One kilo of carbon requires 11.6 kilos of air to produce carbon dioxide.

Thus, the oxygen required 2.66 kilos, which combined with 8.94 kilos of nitrogen (the proportion of oxygen and nitrogen in air) gives 11.6 kilos of air or 9.5 cubic meters.

$$\begin{array}{l}\text{1.0 kilo carbon}\\ \text{11.6 kilos air}\\ \hline \text{12.6}\end{array}\qquad \left.\begin{array}{r}\text{Carbon 1.00 kilo}\\ \text{air}\left\{\begin{array}{l}\text{Oxygen 2.66 kilos}\\ \text{Nitrogen 8.94 ''}\end{array}\right.\\ \hline \text{Total 12.60 ''}\end{array}\right\}\begin{array}{l}\text{Products of}\\ \text{combustion}\end{array}=\left\{\begin{array}{l}\text{3.66 kilos } CO_2\\ \text{8.94 '' } N\\ \hline \text{12.60 ''}\end{array}\right.$$

One kilo hydrogen requires for combustion 34.8 kilos of air, or 28.58 cubic meters:

$$\begin{array}{l}\text{1.0 kilo hydrogen}\\ \text{34.8 kilos air}\\ \hline \text{35.8 ''}\end{array}\qquad \left.\begin{array}{r}\text{Hydrogen 1.0 kilo}\\ \text{air}\left\{\begin{array}{l}\text{Oxygen 8.0 kilos}\\ \text{Nitrogen 26.8 ''}\end{array}\right.\\ \hline \text{35.8 ''}\end{array}\right\}\begin{array}{l}\text{Products of}\\ \text{combustion}\end{array}=\left\{\begin{array}{l}\text{9. kilos } H_2O\\ \text{26.8 '' } N\\ \hline \text{35.8 ''}\end{array}\right.$$

In a similar manner it it found that one kilo of sulphur requires 4.35 kilos of air to produce sulphur dioxide, or 3.6 cubic meters.

The amount of air required for the combustion of one kilo of the coal will be :

COMBUSTIBLES IN THE COAL.

Carbon = 86.49 per cent. = 10.2 kilos of air or 8.32 cubic meters.
Hydrogen = 2.36 " " = 0.82 " " " " 0.67 " "
Sulphur = 0.18 " " = 0.007 " " " " 0.003 " "

One kilo of the coal requires 11.027 " " " " 8.993 " "

or one pound of the coal would require 11.027 pounds or 144.9 cubic feet of air at 62° F. for its combustion.

In a similar manner the amount of air required for the combustion of one kilo of coke (partial analysis given on page 115) would be:

Carbon 94.43 × 11.6 = 10.95 kilos of air, or 8.97 cubic meters, equivalent to 144.4 cubic feet of air per pound of the coke.

The evaporative power of a coal or coke expressed in kilos of water evaporated per kilo of coal, is determined by dividing the total heat of combustion of one kilo of the combustible by 620, which is the total heat (degrees *C.*) of one kilo of steam at atmospheric pressure, raised from water supplied at 62° F. or 16.67° C., or by 536.5 (degrees *C.*) if the water is supplied at 100° C.

If the results are stated in pounds of water evaporated per pound of fuel, it is obtained by dividing the total heat of combustion in "B. T. U." by 1116.6° F., which is the total heat of atmospheric steam raised from water supplied at 62° F., and by dividing by 956.7° F. when the water is supplied at 212° F.

The evaporative value of one kilo of the coal will therefore be, theoretically, assuming the water to be supplied at 16.67° C. (62° F.), and the steam generated at atmospheric pressure:

Carbon, $86.49 \times 8140 \div 100 =$ 7040.28 calories.
Hydrogen, $(2.36 - \frac{O}{8} \times 34500.) \div 100 =$ 695.17 "
Sulphur, $0.18 \times 2220 \div 100 =$ 4.00 "

7739.45 "

$7739.45 \div 620 = 12.48$ kilos of water evaporated per kilo of coal.

	Air required at 16.67°C.	Total heat of combustion.	Equivalent evaporation power of one kilo, and one pound of combustible, under one atmosphere at 100° C.	
			Water at 16.67° C.	Water at 100° C.
One kilo of carbon burning to CO_2	9.5 cubic meters	8140 calories	13.1 kilos	15.2 kilos
One pound of carbon burning to CO_2	152 cubic feet	14650[1] "B.T.U."	13.1 pounds	15.2 pounds
One kilo of carbon burning to CO	4.8 cubic meters	2492 calories	4.0 kilos	4.6 kilos
One pound of carbon burning to CO	76.0 cubic feet	4485 "B.T.U."	4 pounds	4.6 pounds
One kilo of hydrogen burning to H_2O (condensed)	28.5 cubic meters	34500 calories	55.6 kilos	64.3 kilos
One pound of hydrogen burning to H_2O ,,	457 cubic feet	62100 "B.T.U."	55.6 pounds	64.3 pounds
One kilo of sulphur burning to SO_2	3.6 cubic meters	2220 calories	3.5 kilos	4.1 kilos
One pound of sulphur burning to SO_2	57 cubic feet	3996 "B.T.U."	3.5 pounds	4.1 pounds

[1] The figure 14500 "B. T. U." is in general use, 14650 "B. T. U." however, being nearer correct.

If the water be supplied at 100° C., the evaporative value will be $7739.45 \div 536.5 = 14.42$ kilos of water evaporated per kilo of coal.

The actual evaporation is less, in boiler practice, than the theoretical as computed above, for the following reasons:

1. There may be a loss due to incomplete combustion.
2. There is necessarily a considerable amount of heat carried off by the chimney gases.
3. There is loss of heat due to radiation.
4. Heat is also lost, due to the evaporation of the hydroscopic moisture contained in the coal and to the heat in the vapor formed by the combustion of the hydrogen in the coal.

For example, in a test of a standard type of boiler made by Prof. J. E. Denton, where the fuel, anthracite coal, was burned so thoroughly as to practically eliminate the loss due to incomplete combustion, the remaining losses were as follows:

Loss of heat by chimney	13.83	per cent.
" " " " radiation	2.64	" "
" " " " moisture	0.08	" "
Total	16.55	" "

These per cents being in terms of total heat per pound of combustible. Consult article on Boiler Tests.

The total heat as determined by calorimetric measurements being 14302. "B. T. U." per pound of combustible.

The heat imparted to the steam was $100 - 16.55 = 83.45$ per cent. of the total heat.

This is a high economical result. Ordinarily the heat imparted to the steam is not over 80 per cent. of the total heat, so that the *available* heat is usually less than 80 per cent. of the theoretical heat.

Calorimetry.

Of the many instruments in use in calorimetry for determining the heating power of coals, the Mahler, the Thompson, the Barrus, and the Carpenter are selected for description.

For rapidity and accuracy, the Mahler is to be recommended.

This apparatus consists of a modified form of Berthelot's bomb.

Berthelot's instrument, which was originally made for the combustion of gases under pressure, consisted of a steel cylinder lined with platinum.

Mahler uses porcelain as a lining to the steel cylinder in place of the platinum, thereby materially reducing the cost of the apparatus.

The accompanying sketches represent a vertical section of the calorimeter itself, showing all of the attachments, and also a vertical section of the shell to a larger scale. The shell is forged out of mild steel having a tensile strength of thirty-one tons to the square inch, and an elongation of twenty-two per cent. It is about eight millimeters thick and usually weighs about 3,500 grams, with a capacity of 814.6 cc. The capacity of the instrument was made much greater than that of M. Berthelot for two reasons: First, to insure complete combustion, and second, because many gaseous fuels used for industrial purposes contain nitrogen and carbon dioxide. It is necessary to take a large quantity of them in order to obtain a measurable rise in temperature. The shell is coated on the inside with porcelain to protect it from corrosion or oxidation. The porcelain being very thin, does not interfere with the transmission of heat. The cover is fitted with a ferro-nickel cock R, with a conical screw and stuffing box E, for the introduction of oxygen under pressure. The cover is screwed down upon a ring of lead P, placed in a circular groove cut in the rim of the shell, making a tight joint. Through the cover passes an isolated electrode, to which a platinum rod is fastened by means of a clamp. Another platinum rod is fastened to the cover, and the pan which contains the substance to be burned is attached to this by means of another platinum rod and two clamps. Attached to the platinum rods and passing through the substance to be burned is a small helix of fine iron wire. Ignition is produced by heating this wire white-hot by

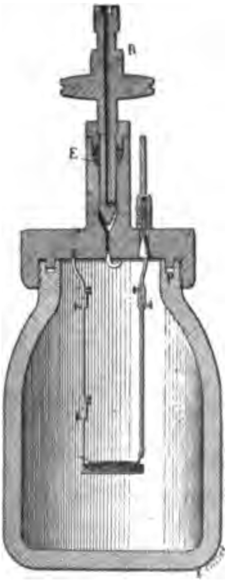

Fig. 33.

means of a battery. The calorimeter, the outer vessel, and the various details of M. Mahler's apparatus differ from the analogous parts of M. Berthelot's instrument. The calorimeter is of thin brass and contains about 2.3 kilos of water. The large amount of water practically eliminates all error due to evaporation. The agitator S is worked by the lever L, which pushes

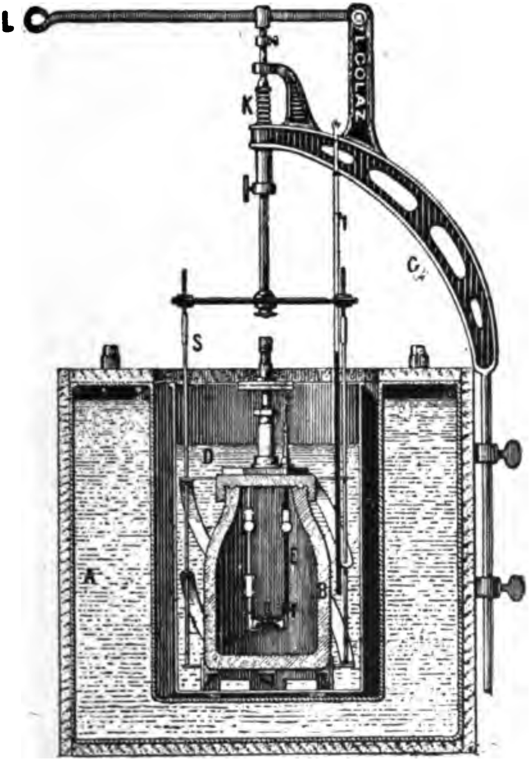

Fig. 34.

down the rod K, to which the agitator is attached. This rod has a spiral thread on it and moves through a nut, so that in pressing it down it also receives a revolving motion, thus very thoroughly stirring the water. The thermometer T should read to the one hundredth of a degree. For igniting the substance a battery capable of giving a current of two amperes with an E.

M. F. of ten volts is required. The oxygen is supplied in cylinders of 125 cubic feet capacity at a pressure of 150 atmospheres. Such a cylinder will supply oxygen enough for about 140 determinations.

Before this instrument can be used for determining calorific power, it is necessary to find the water equivalent of the shell and its appendages. This must be determined with the utmost care, as upon it depends the correctness of all the results afterward obtained. It may be calculated directly from the weights and known specific heats of the parts. It may also be obtained experimentally. The method by calculation can only be approximate, because of the weight of the porcelain of the shell is not known and can only be estimated. This method gives the following results :

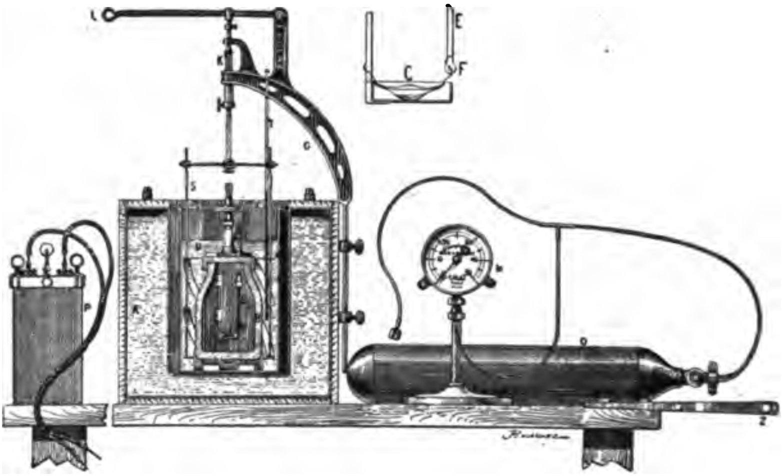

Fig. 35.

Material.	Weight in grams.	Specific heat.	Water equivalent in grams.
Brass of calorimeter	703.07	0.094	66.088
Steel of calorimeter	3,323.25	0.1165	387.157
Porcelain	134.078	0.179	24.0
Platinum....................	21.3	0.0324	0.68
Lead	9.0	0.0314	0.282
Glass of thermometer	12.69	0.17968	3.114
Mercury of thermometer ...	25.03	0.03332	0.833
Oxygen	29.1205	0.155	3.513
			485.657

In determining the water equivalent the following method is employed. The shell is charged with oxygen at twenty-five atmospheres pressure.

A known weight of water, about 2000 grams, is then placed in the calorimeter, the shell immersed in it, and the whole apparatus placed under the same conditions that would exist during an actual combustion. The water is then agitated until the temperature becomes constant, when about 300 grams of water at a much lower temperature are added, and the whole agitated until the temperature again becomes constant. Readings of the thermometer are taken every half minute. From the observed fall of temperature the water equivalent may be calculated by means of the following formula:

Let X = water equivalent of calorimeter shell and appendages.
t = final temperature of water in calorimeter.
t_1 = initial " " " " "
t_2 = initial temperature of cold water added.
W = weight of water in calorimeter at beginning of experiment.
w = weight of cold water added.

Then we have:

$$(t_1 - t) W + (t_1 - t) X = (t - t_2) w,$$

Or,

$$X = \frac{(t - t_2) w - (t_1 - t) W}{t_1 - t}.$$

The results of twenty-five determinations gives a mean of 489.97, or practically 490.

Method of Making a Determination with Coal.

About ten grams of coal to be tested is finely powdered and passed through a sieve having 10,000 meshes to the square inch. It is necessary that the coal be very fine or it will not burn completely. The powdered sample is placed in a glass weighing tube and carefully weighed. The platinum wires and pan are attached to the cover of the shell and the iron wire helix placed in position. A sample of the coal is now poured into the pan from the weighing tube, its weight determined, care being taken to see that none is spilled and that the iron wire helix passes through the coal. The cover is then placed on the shell and screwed down firmly. The shell is now connected with the oxygen cylinder, and the oxygen allowed to flow in until the gauge shows a pressure of about twenty-five atmospheres. The stop-cock is then closed and the shell placed in the calorimeter, which has been previously partially filled with about 2,400 grams of water. The thermometer and agitator are adjusted, and the whole well stirred to obtain a uniform temperature. The temperature is then observed, from minute to minute, for four or five minutes, so as to determine its rate of change. The charge is then ignited by connecting one pole of the battery to the electrode F, and touching the other pole to any part of the shell. The temperature is observed each minute until it begins to fall regularly, and then each minute for five minutes in order to ascertain the law of cooling. The agitator should be kept going constantly during the whole period of the observation. The shell is now removed from the water, the stop-cock R opened to let out the gas, and then the shell itself is opened. The shell should be rinsed out with distilled water to collect the acid formed during combustion. The amount of acid carried out with the escaping gas is negligible. The calorific power of the coal may now be calculated as follows:

Let Q = calorific power of the coal.
Δ = observed rise of temperature.
x = correction for radiation.
P = weight of water taken in grams.
P' = water equivalent of shell, appendages, and gas.
p = weight of nitric acid found.
p' = weight of iron wire helix.
0.23 calorie = heat of formation of one gram of nitric acid.
1.6 calories = heat of combustion of one gram of iron.

Then

$$Q = (\Delta + x)(P + P') - (0.23p + 1.6p').$$

Example Showing Method of Calculation.

1.042 gram of coal was taken.

The calorimeter contained 2,276.6 grams of water.

The water equivalent of apparatus = 490 grams.

The pressure of oxygen = 25 atmospheres.

The law of variation of temperature in the calorimeter before combustion is expressed by $X_0 = O$.

The law of variation during subsequent period is

$$X_1 = 27.46 - 27.395 = 0.065^\circ \text{ C.}$$

Hence, during the period of combustion the system lost 0.065 degree by radiation.

The apparent variation of temperature is

$$27.460 - 24.855^\circ = 2.605^\circ \text{ C.}$$

Actual variation $= 2.605^\circ + 0.065^\circ = 2.67^\circ$ C.

The nitric acid formed = 0.15 gram. And the weight of iron wire = 0.025 gram. Hence, heat of formation of nitric acid = $0.15 \times 0.23 = 0.0345$ calorie, and heat of combustion of wire = $0.025 \times 1.6 = 0.04$ calorie. Heat of combustion of coal = $2.67 \times (2,276.6 + 490)$,

$$\begin{aligned} &= 7,386.8 \text{ calories.} \\ 7,386.6 - (0.0345 + 0.04) &= 7,386.72 \quad \text{“} \\ + 1.042 &= 7,088.9 \quad \text{“} \end{aligned}$$

7088.9 calories per kilo. = 12760. B. T. U. per pound of coal.

To show the accuracy with which this calorimeter works, five samples of willow charcoal were burned, with the following results:

Average of five determinations	7973	calories per kilo.
Highest determination	7975	“ “ “
Lowest determination	7971	“ “ “

Five determinations of a sample of bituminous coal from Coleman County, Texas, gave as follows:

Average of five determinations	6766.0	calories per kilo.
Highest determination	6793.6	“ “ “
Lowest determination	6720.3	“ “ “

References.—“On the Berthelot-Mahler Calorimeter for the Calorific Power of Fuels.” Prof. A. M. Mayer. *Stevens' Indicator*, April, 1895, p. 133–148.

“Zur Werthbestimmung der Brennstoffe.” (Verfahren und Calorimeter von Mahler, Bunte, Fischer, Scheurer-Kestner), *Stahl und Eisen*, 13, 52.

Determination industrielle du pouvoir calorifique des combustibles. Mahler. *La Sucrerie Indigène*, 41, 443.

THE THOMPSON CALORIMETER.

This instrument, in general use in England for calorimetric determinations of solid fuels, is shown in Fig. 36.

Fig. 36.—Thompson Calorimeter.

The water equivalent (theoretical) of the calorimeter is found by weighing each part carefully and multiplying by its specific heat.

Thus:

	Weight.			Specific heat.		Water equivalent.
Part of glass cylinder in contact with the water	922.43	grams	×	0.19768	=	182.245
Glass bell jar	75.381	"	×	0.19	=	14.313
Brass base	99.853	"	×	0.09391	=	9.377
Four copper disks........	65.100	"	×	0.09515	=	6.294
Brass over top of bell jar.	21.307	"	×	0.09391	=	2.001
Copper tube..............	30.800	"	×	0.09512	=	2.930
Rubber cork	1.578	"	×	0.331	=	0.552
Rubber tube	1.784	"	×	0.331	=	0.591
Platinum crucible	15.111	"	×	0.324	=	0.490
Mercury of thermometer.	9.583	"	×	0.333	=	0.319
Glass of thermometer	7.350	"	×	0.19	=	1.396
Total						220.478

This theoretical water equivalent should be checked by a determination by direct experiment, as follows:

The calorimeter is taken and adjusted under the conditions of use.

2000 grams of distilled water are weighed out and the temperature taken: call this temperature t.

Let t_1 = temperature of the apparatus.

The 2000 grams of water are poured into the glass cylinder *ab*, Fig. 36, the other parts c, d, g, h, etc., placed in position inside the cylinder, and the water kept well stirred by means of the discs K. K. on the side of the bell jar.

After agitating it about fifteen minutes (about the time required for a coal combustion) the temperature is taken; this temperature call t_0. To correct for radiation it is necessary to continue this operation for an equal period of time, calling the last temperature c, from which we obtain the fall of temperature to be $(t_0 - c) = r$. Expressing this in a formula

$$\frac{2000\ (t - (t_0 + r)}{(t_0 + r) - t_1} = \text{water equivalent}$$

r being the fall of temperature due to radiation.

Thus:

Temperature of apparatus = 14.6° C.
" " water = 19.5° C.
Final " " " = 18.65° C.

Correction $(18.65 - 18.3) = 0.35$.

$$\frac{2000\ (19.5 - 19.0)}{19 - 14.6} = 227.22.$$

By calculation the water equivalent is 220.47.
" experiment " " " " 227.22.

The combustion with a sample of coal is performed as follows:

An incandescent paper (about one mm. long) is dropped into the crucible (f) containing one gram of the *very finely* pulverized coal, the oxygen supply being slowly turned on and the inverted bell jar (t) containing the crucible (f) is gently lowered into the 2000 grams of water contained in the glass cylinder (*ab*).

The combustion will be quite active: the gaseous products will bubble through the water and give up their sensible heat.

After the fuel has been consumed the supply of oxygen is stopped and the glass tube c is opened, permitting the water to enter the bell jar and flow into and submerge the crucible so that the whole of the apparatus and water is raised to a uniform temperature.

It will be noted that the coal burns gently at first. The oxygen introducing pipe (*g h*) should not be projected too low into the bell jar until the volatile hydrocarbons are consumed; the residual fixed carbon is more difficult to burn.

The oxygen supply tube should consequently be projected so as to deliver the oxygen immediately over the platinum crucible, and to more effectually burn the fuel, the tube may be slightly rotated.

Great care must be taken in reading the thermometer before and after the gram of coal is burned: the difference of these two readings gives the rise in temperature for the amount of coal taken, which when multiplied by 2000 plus the water equivalent of the calorimeter, gives the heating power of the coal.

But since heat is being radiated to the air during the experiment, a correction must be made. To determine this, it is necessary to note the time required to burn the coal, and then agitate the apparatus for a corresponding period. During this last agitation the temperature will fall somewhat; this fall, divided by two, will give tho proper correction.

The figure obtained is an average of the whole radiation: should the fall be taken direct, it would give the correction for radiation when the water is at its maximum temperature. The following is the analysis of a sample of coal, the theoretical heating power calculated from the analysis, and calorimetric determination of the coal by means of the Thompson calorimeter, and a comparison of the number of calories per kilo derived by calculation and by direct experiment.

Analysis:

Carbon	84.80	per cent.
Hydrogen	2.42	"
Sulphur	0.62	"
Nitrogen	0.93	"
Moisture	1.03	"
Oxygen	3.19	"
Ash	7.01	"
Total	100.00	"

the theoretical heating value being:

$$\frac{(8140 \times 84.8) + (34500 \times 2.42 - 0.4) + 2220 \times 0.62}{100} = 7513$$

calories per kilo of coal.

The test of the coal by the Thompson calorimeter gave as follows:

Amount of coal taken = 0.445 gram.
Temperature of water and apparatus (initial)...... 18.95° C.
Maximum temperature " " .... 20.45° C.
Final temperature (used for correction of radiation) 20.40° C.

$$\text{Correction for radiation} \left\{ \frac{20.45 - 20.40}{2} = 0.025^\circ \text{ C.} \right.$$

Rise in temperature for 0.445 gram = 1.525° C.
" " " " 1.000 " = 3.43° C.
2227 × 3.43 = 7638.6 calories per kilo of coal.

THE BARRUS COAL CALORIMETER.[1]

The complete apparatus is shown in the accompanying figure (37). The calorimeter itself consists of a glass vessel five inches in diameter, nine and a half inches high, which holds the water of the calorimeter. Submerged in the interior is a bell-shaped glass vessel two and a half inches in diameter, four inches high, having a long neck three-fourth of an inch in diameter, which is closed at the top with a stopper.

The upper end of the neck stands five inches above the top of the outside vessel. The glass bell, or "combustion chamber," as it may be termed, rests upon a metal base, to which it is held by means of spring clips, the bottom of the chamber being provided with an exterior rib by means of which the clips are made fast. The base is perforated, and at the center is mounted a short tube, for the reception of a crucible in which the combustion takes place. The crucible is made of platinum. It is surrounded by a layer of non-conducting material, which is placed between it and the outer metal. A small glass tube is inserted in the stopper at the top of the neck, and this is carried down to the interior of the combustion chamber. It is fitted somewhat loosely, so that a slight pressure will move it up or down, and thereby adjust its lower end to any height desired above the crucible. The tube has a slight lateral movement also, so that

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[1] Transactions American Society Mechanical Engineers, 14, 816.

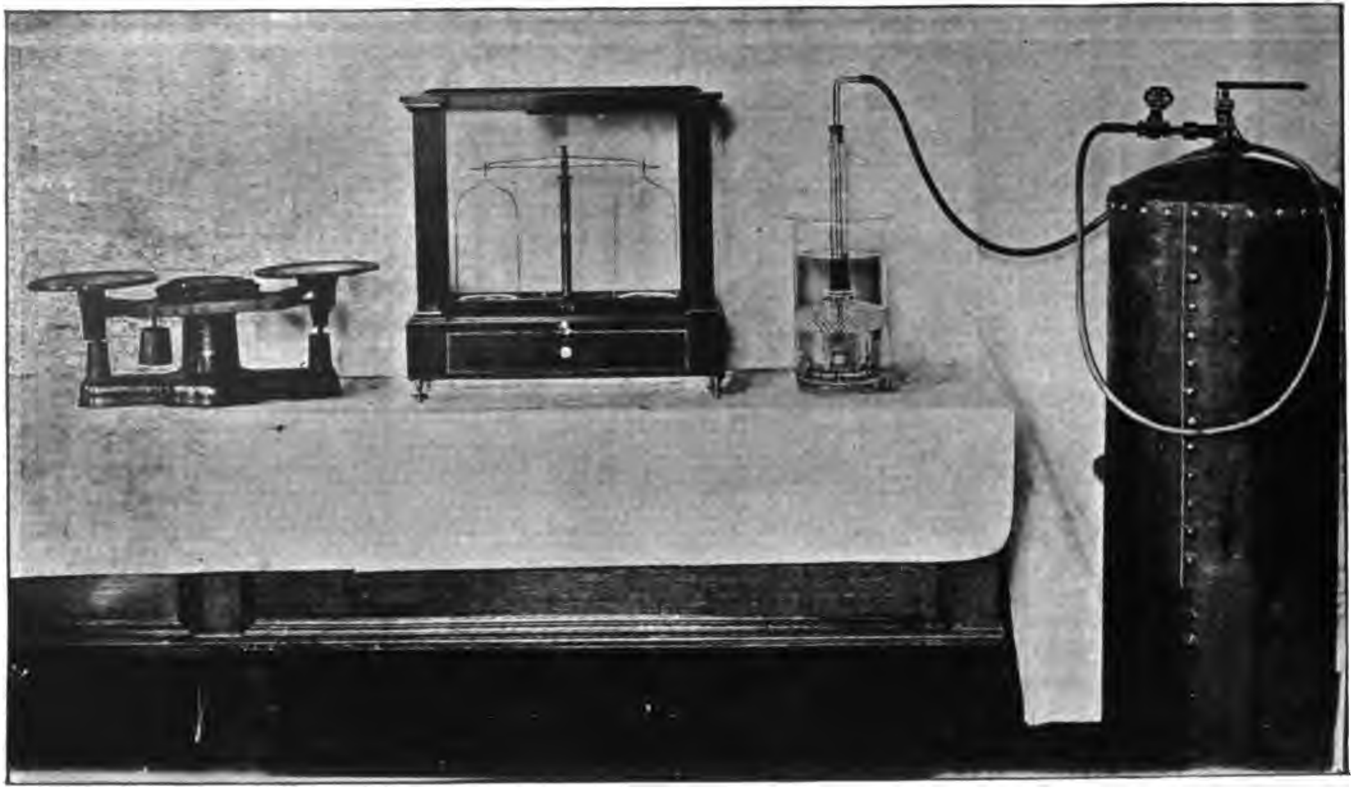

Fig. 37.

it may be directed, at the will of the operator, toward any part of the crucible.

This tube is connected with a tank containing oxygen gas, and through it a current of gas is passed, so as to enable the combustion of the coal to be carried on under water.

The pressure of the gas drives out the water which would otherwise fill the chamber, and keeps its level between the base. The products of combustion rising from the crucible pass downward through the perforations in the base, escaping around the edge of the base, and finally bubbling up through the water and emerging at its surface. A wire screen is secured to the neck of the combustion chamber, extending to the sides of the outer vessel, thereby holding back the gas and preventing its immediate escape to the surface of the water.

In making the test the quantity of water used is 2000 grams and the quantity of coal one gram. The equivalent colorific value of the material of the instrument is 185 milligrams (0.185 gram).

One degree rise of temperature of the water corresponds, therefore, to a total heat of combustion of 2185 B. T. U. The number of degrees rise of temperature for ordinary coals varies from 5.5° to 6.5° F.

The thermometer used for determining the temperature of the water is graduated to twentieths of a degree; and as the divisions are about one-thirtieth of an inch apart, they may be subdivided by the eye so as to readily obtain a reading to hundredths of a degree.

The scales shown at the extreme left of the cut are used for weighing out the water, and the chemical scales shown in the center are employed in weighing the coal and ash.

The process of making a test is as follows:

Having dried and pulverized the coal, and weighed out the desired quantities of coal and water, the combustion chamber is immersed in the water for a short time, so as to make the temperature of the whole instrument uniform with that of the water. On its removal the initial temperature of the water is observed, the top of the chamber lifted, the gas turned on, and the coal quickly lighted, a small paper fuse having been previously inserted in the crucible for this purpose. The top of the combustion chamber is quickly replaced, and the whole returned to its submerged position in the water. The combustion is carefully watched as the process goes on, and the current of oxygen is directed in such a way as to secure the desired rate and conditions for satisfactory combustion. When the coal is entirely consumed, the interior chamber is moved up and down in the water until the temperature of the whole has become uniform, and finally it is withdrawn and the crucible removed. The final temperature of the water is observed, and the weight of the resulting ash.

The initial temperature of the water is so fixed by suitably mixing warm and cold water that it stands at the same number of degrees below the temperature of the surrounding atmosphere (or approximately the same) as it is raised at the end of the process above the temperature of the air. In this way the effect of radiation from the apparatus is overcome so that no provision in the matter of insulation is required, and no allowance needs to be made for its effect.

RESULTS OF TESTS WITH THE BARRUS COAL CALORIMETER.

Cumberland Coals.

Number for reference.	Kind of coal : Mine or locality	Percentage of ash.	Total heat of combustion.
1		7.6	13,868 B. T. U.
2		8.2	14,058 " " "
3	George's Creek	6.1	14,217 " " "
4	" "	6.6	13,925 " " "
5	" "	8.6	12,874 " " "
6	" "	6.5	12,921 " " "
7	" "	7.0	13,360 " " "
8	" " (American Co.)	5.0	13,487 " " "
9	" " (Md. Coal Co.)	5.1	13,656 " " "
10	" " (G. C. Coal and Iron Co.)	5.7	13,424 " " "
11		6.1	13,534 " " "
12	George's Creek	5.1	13,745 " " "
13		7.5	13,617 " " "
14	Eureka	5.1	13,653 " " "
15	"	5.4	13,427 " " "
16		8.	12,973 " " "
17	George's Creek	4.4	13,923 " " "

Fischer's calorimeter, while somewhat more complex than the Mahler or Thompson's, is an accurate instrument for the determination of the heating power of fuels. Consult *Chemische Technologie der Brennstoffe*, von Dr. Ferdinand Fischer, p. 401-

CARPENTER'S COAL CALORIMETER.

R. C. Carpenter[1] has devised a calorimeter for the determination of the heating power of coals, which is thus described. The general appearance of the instrument is shown in Fig. 38, a sectional view of the interior is shown in Fig. 39, from which it is seen that, in principle, the instrument is a large thermometer, in the bulb of which combustion takes place, the heat being absorbed by the liquid which is within the bulb. The rise in temperature is denoted by the height to which a column of liquid rises in the attached glass tube.

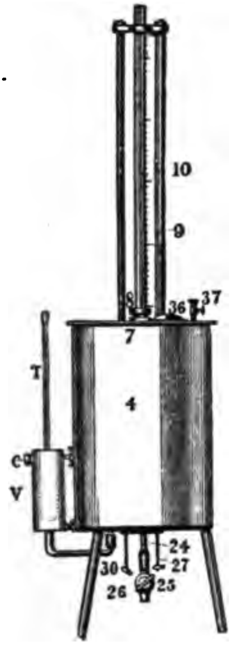

Fig. 38.

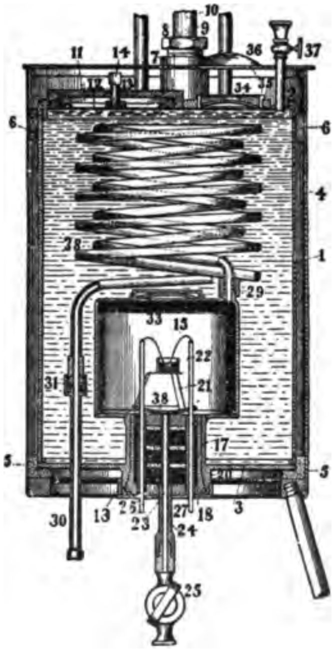

Fig. 39.

In construction, Fig. 39, the instrument consists of a chamber,

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[1] Transactions Amer. Society of Mechanical Engineers, Vol. XVI, (June, 1895.)

No. 15, which has a removable bottom, shown in section in Fig. 39 and in perspective in Fig. 40. The chamber is supplied with oxygen for combustion through tube, 23, 24, 25, the products of combustion being discharged through a spiral tube, 29, 28, 30.

Surrounding the combustion chamber is a larger closed chamber, 1, Fig. 38, filled with water, and connecting with an open glass tube, 9 and 10. Above the water chamber, 1, is a diaphragm, 12, which can be placed in position by screw, 14, so as to adjust the zero level in the open glass tube at any desired point. A glass for observing the process of combustion is inserted at 33 in top of the combustion chamber, and also at 34 in top of the water chamber, and at 36 in top of outer case.

This instrument readily slips into an outside case, which is nickel plated and polished on the inside, so as to reduce radiation as much as possible. The instrument is supported on strips of felting, 5 and 6, Fig. 39. A funnel for filling is provided at 37, which can also be used for emptying, if desired.

The plug which stops up the bottom of the combustion chamber carries a dish, 22, in which the fuel for combustion is placed; also two wires passing through tubes of vulcanized fiber, which are adjustable in a vertical direction and connected with a thin platinum wire at the ends. These wires are connected to an electric current and used for firing the fuel. On the top part of the plug is placed a silver mirror, 38, to deflect any radiant heat. Through the center of this plug passes a tube, 25, through which the oxygen passes to supply combustion. The plug is made with alternate layers of rubber and asbestos fiber, the outside only being of metal, which, being in contact with the wall of the water chamber, can transfer little or no heat to the outside.

The discharge gases pass through a long coil of copper pipe, and are discharged through a very fine orifice in a cap at 30.

The instrument has been so designed that the combustion can take place in oxygen gas having considerable pressure, but in pressure it has been found that very excellent results have been obtained with pressures of two to five pounds per square inch, and these having been commonly used in the determinations.

Two instruments have been built at the present time, which differ from each other somewhat in detail, but principally in dimensions. The first instrument held about one pound of water, and was intended for use with about one gram of coal. In that instrument the entire bottom of the water chamber was removable and the whole of the combustion chamber. This form, while giving fully as good results as the one described, was more likely to leak, and, consequently, was difficult to keep in good condition. The first form built employed an adjusting piston to regulate the initial heading of the water column, which, possibly, may have been as good as ,the diaphragm used at present.

The instrument described, which is of later design, holds about five pounds of water, and is large enough for the consumption of two grams of coal.

Full details for manipulation of the apparatus are given in *Trans. Amer. Society Mechanical Engineers*, Vol. XVI, (1895).

Fig. 40.

References.—"Uber die Bestimmung des Heizwerthes der festen Brennmaterialien und Bericht über die wichtigere neure Litteratur dieses Gebietes;" von Knorre, *Die Chemische Industrie*, 17, 93.

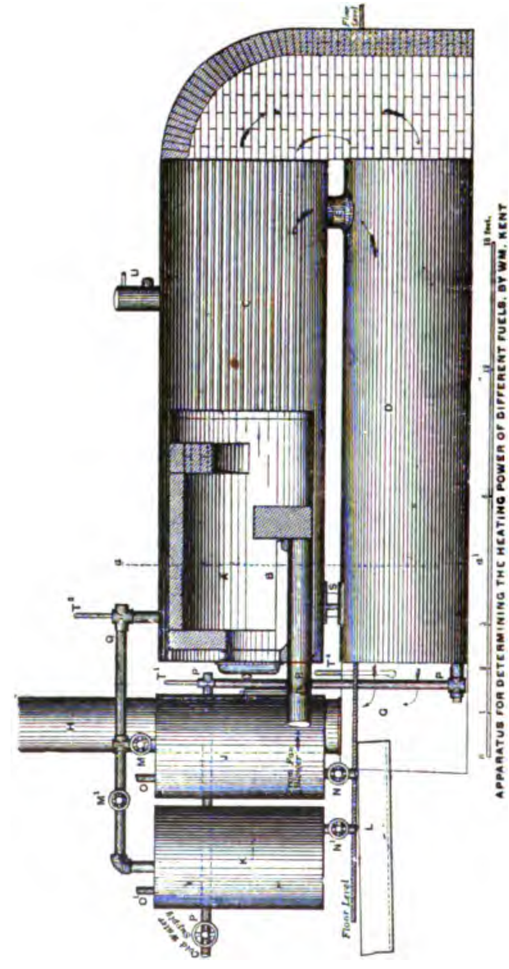
"Etude sur les combustibles et la combustion," Vivien, *La Sucrarie indigène*, 44, 261.

Determination of the heating power of coal by the use of large amounts of coal either (a) in specially constructed apparatus for the same, or (b) under boilers in actual practice.

Apparatus for determining the heating value of Fuel, by Wm. Kent, M.E., (Fig. 41).

Its principal feature is that it is not a steam boiler but a water heater. It consists of two sheet-metal cylinders, each twelve feet long, the upper one four feet in diameter and the lower one three feet, and connected by a short neck at one end only.

The upper cylinder is provided with a fire-box three and a half feet in diameter and six feet long, and its rear end is filled with about 100 two-inch tubes. The lower cylinder is completely filled with two-inch tubes. The fire-box is lined throughout with fire-brick, and contains a grate surface two feet wide by two and a half feet long. A hanging bridge-wall of fire-brick is placed in the upper part of the fire-box in the rear of



APPARATUS FOR DETERMINING THE HEATING POWER OF DIFFERENT FUELS. BY WM. KENT

Fig. 41.

A, Fire box.
B, Grate bars.
C, Tubes of upper heater.
D, Lower heater.
E, Connection between heaters.
F, Flue between heaters.[1]
G, Chimney connection.
H, Chimney.
J K, Water-measuring tanks (closed tops.
L, Waste drain for hot water.
M M¹, Admission valves to tanks.
N N¹, Emptying valves.
O O¹, Overflow and vent-pipes.
P, Cold water supply-pipe.
Q, Hot water outlet.
R, Air-supply pipe.
T¹T², Thermometers for water.
T³T⁴, Thermometers for air and waste gas.
S, Support of upper heater.
U, Safety valve.

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[1] A water-jacketed iron flue can be substituted for the brick one with less radiation of heat.

the bridge-wall proper, for the double purpose of presenting a hot fire-brick surface to the flame before allowing it to touch the heating surfaces of the tubes and tube-sheet, and of changing its direction so as to cause the gases to thoroughly commingle, and thus to insure complete combustion. In testing highly bituminous coals, it might be advisable to have more than one of these hanging walls, and to give the fire-box a greater length, to more certainly insure complete combustion of the gases. The gases of combustion pass through the tubes of the upper heater, then down through a fire-brick connection into the tubes in the lower heater, after leaving which they pass into the chimney. Air is fed to the fire, under the grate-bars, through a pipe leading from a fan-blower. The air is measured by recording the revolutions of the blower, and the measurement is checked by an anemometer in the air-pipe. Its weight should be calculated from the barometric pressure, and its contained moisture should also be determined. Its temperature should be taken before it enters the ash-pit.

The temperature of the escaping gases should be taken by several thermometers, the bulbs of which reach to different portions of the chimney connection. Cold water is supplied to the bottom of the lower heater, at the chimney end, its temperature being taken before it enters by a thermometer inserted in the pipe. The water supply pipe may be conveniently attached to the city main. The water passes through the two heaters in an opposite direction to that of the gases of combustion, and escapes at the outlet pipe at the top of the upper heater by which it is taken to two measuring tanks, which are alternately filled and emptied. The temperature of the outflowing water is taken by a thermometer inserted in the overflow pipe. The rate of flow of water through the apparatus is regulated so that the temperature of the outflowing water does not exceed 200° F. The measuring tanks have closed tops, which prevent evaporation, small outlet pipes being attached to the top of each, which serve both as indicators when the tanks are full, and to allow air to escape from the tank when it is being filled with water.

The grate surface being only five square feet and the heating surface about 1000 square feet, the ratio of 200 to 1, or more than five times the usual proportion in a steam boiler, and the water being much colder than that in a steam boiler, the gases of combustion should be cooled down to near the temperature of the air supplied to the fire, especially when, as is usually the case, the water supply is colder than the air. For extremely accurate tests, the water might be cooled before entering by a refrigerating apparatus or by ice.

The whole apparatus being thoroughly protected by felting from radiation, the heat generated by the fuel is all measured in the increase of heat given to the water which flows through the apparatus, and in the increase of temperature of the gases of combustion as taken in the chimney, over the temperature of the air supplied to the fire. This increase, however, being in any case very slight, and the quantity of air being known, the amount of heat from the fuel which escapes up the chimney can be calculated with but small chances of error.

Boiler Test.

RÉSUMÉ OF TESTS UPON BABCOCK & WILCOX BOILERS.[1]

Name of coal.	Duration of test hours.	Grate surface, square feet.	Heating surface, square feet.	Percentage of refuse.	Coal burned per square ft. of grate, pounds.	Water evaporated per square foot of heating surface per hour, pounds.	Water per pound, coal, from and at 212° F. pounds.	Water per pound combustible from and at 212° F.[2]	Rated horse power.	Horse power developed.
Anthracite scr's ⅕. Powton, Pa. Semi-bitum ⅘.	10¼	60.0	3126	8.8	17.6	4.32	11.32	12.42	272	448
Jackson, O., nut	8	48.0	3358	9.6	32.1	4.11	8.93	9.88	262	460
Castle Shan'n Pa. ⅜ nut. ⅝ lump.	42¼	69.1	4784	10.5	27.9	4.13	10.00	11.17	416	570
Cardiff, lump ..	6¾	21.2	1564	11.7	26.7	3.69	10.07	11.40	136	189

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[1] Trans. Amer. Soc. Mechan. Engineers, 4, 267.

[2] The term "per pound of combustible" represents one pound of the heating constituents of the coal, viz.: ashes and moisture taken out.

APPROXIMATE HEATING VALUE OF COALS. (KENT.)

Percentage of fixed carbon in coal dry and free from ash.	Heating value "B. T. U." per pound of combustible.	Equiv. water evaporated from and at 212° F. per pound of combustible.	Percentage of fixed carbon in coal dry and free from ash.	Heating value of "B. T. U." per pound of combustible.	Equiv. water evaporated from and at 212° F. per pound of combustible.
100	14500	15.00	68	15480	16.03
97	14760	15.28	63	15120	15.65
94	15120	15.65	60	14580	15.09
90	15480	16.03	57	14040	14.53
87	15660	16.21	54	13320	13.79
80	15840	16.40	51	12600	13.04
72	15660	16.21	50	12240	12.67

The use of the table may be shown as follows:

Given a coal containing moisture two per cent., ash eight per cent., fixed carbon sixty-one per cent., and volatile combustible matter twenty-nine per cent., what is its probable heating value?

Deducting moisture and ash we find the fixed carbon is 61.90 or sixty-eight percent. of the total fixed carbon and volatile combustible matter.

One pound of coal dry and free from ash would, by the table, have a heating value of 15480 thermal units, but as the ash and moisture having no heating value, are ten per cent. of the total weight of the coal, the coal would have ninety per cent. of the table value, or 13932 thermal units. This divided by 966, the latent heat of steam at 212° F., gives an equivalent evaporation per pound of coal of 14.42 pounds.

The heating value that can be obtained in practice from this coal would depend upon the efficiency of the boiler, and this largely upon the difficulty of thoroughly burning the volatile combustible matter in the boiler furnace. If a boiler efficiency of sixty-five per cent. could be obtained, then the evaporation per pound of coal from and at 212° F. would be 14.42 × 0.65 = 9.37 pounds.

With best anthracite coal, in which the combustible portion is, say ninety-seven per cent. fixed carbon and three per cent. volatile matter, the highest result that can be expected in a boiler test with all conditions favorable, is 12.2 pounds of water evaporated from and at 212° F. per pound of combustible, which is eighty per cent. of 15.28 pounds, the theoretical heating power.

With the best semi-bituminous coals, such as Cumberland and Pocahoutas, in which the fixed carbon is eighty per cent. of the total combustible, 12.5 pounds, or seventy-six per cent. of the theoretical 16.4 pounds may be obtained.

For Pittsburgh coal, with fixed carbon ratio of sixty-eight per cent., eleven pounds, or sixty-nine per cent. of the theoretical 16.03 pounds, is about the best practically obtainable with the best boilers.

With some good Ohio coals, with a fixed carbon ratio of sixty per cent., ten pounds, or sixty-six per cent. of the theoretical 15.9 pounds has been obtained under favorable conditions, with a fire-brick arch over the furnace with coals mined west of Ohio; with lower carbon ratios, the boiler efficiency is not apt to be as high as sixty per cent.

From these figures a table of probable maximum boiler test results from coals of different fixed carbon ratios may be constructed as follows:

Fixed carbon ratio	97.0	80.0	68.0	60.0	54.0	50.0
Evaporated from and at 212° F. per pound combustible, maximum in boiler tests	15.1	12.5	11.0	10.0	8.3	7.0
Boiler efficiency, per cent	80.0	76.0	69.0	66.0	60.0	55.0
Loss, chimney radiation, imperfect combustion, etc	20.0	24.0	31.0	34.0	40.0	45.0

The difference between the loss of twenty per cent. with anthracite and the greater losses with the other coals is chiefly due to imperfect combustion of the bituminous coals, the more highly volatile coals sending up the chimney the greater quantity of smoke and unburned hydrocarbon gases. It is a measure of the inefficiency of the boiler furnace and of the inefficiency of heating surface caused by the deposition of soot, the latter being primarily caused by the imperfection of the ordinary furnace and its unsuitability to the proper burning of bituminous coal. If in a boiler test with an ordinary furnace lower results are obtained than those in the above table, it is an indication of unfavorable conditions, such as bad firing, wrong proportions of boiler, defective draft, and the like, which are remediable. Higher results can be expected only with gas producers, or other styles of furnace especially designed for smokeless combustion.

The efficiency of a boiler is the percentage of the total heat generated by the combustion of the fuel, which is utilized in heating the water and in generating steam. With anthracite coal the heating value of the combustible portion is very nearly 14500 "B. T. U." per pound, equal to an evaporation from and at 212° F. of $14500 \div 966 =$ fifteen pounds of water. A boiler which when treated with anthracite coal shows an evaporation of twelve pounds of water per pound of combustible has an efficiency of $12 \div 15 = 80$ per cent., a figure which is approximate, but scarcely ever quite reached in the best practice.

With bituminous coal it is necessary to have a determination of its heating power made by a coal calorimeter before the efficiency of the boiler using it can be determined, but a close estimate may be made from the chemical analysis of the coal.

The difference between the efficiency obtained by the test and 100 per cent. is the sum of the numerous wastes of heat, the chief of which is the necessary loss due to the temperature of the chimney gases. If we have an analysis and a calorimetric determination of the heating power of the coal, and an average analysis of the chimney gases, the amounts of the several losses may be determined with approximate accuracy by the method described below. Data given :

1. ANALYSIS OF THE COAL. CUMBERLAND SEMI-BITUMINOUS.

Carbon	80.55	per cent.
Hydrogen	4.50	"
Oxygen	2.70	"
Nitrogen	1.08	"
Moisture	2.92	"
Ash	8.25	"
	100.00	"

2. ANALYSIS OF THE DRY CHIMNEY GAS BY WEIGHT.

		C.	O.	N.
CO_2	13.6	3.71	9.89	....
CO	0.2	0.09	0.11	....
O	11.2	....	11.20	....
N	75.0	....	....	75.0
Total	100.0	3.80	21.20	75.0

The gases being collected over water, the moisture in them is not determined.

Heating value by Dulong's formula = 14243 heat units.

3. Ash and refuse as determined by boiler test 10.25 per cent. or two per cent. more than that found by analysis, the difference representing carbon in the ashes obtained in the boiler test.

4. Temperature of external atmosphere 60° F.

5. Relative humidity of air, sixty per cent. corresponding to 0.007 pound of vapor in each pound of air.

6. Temperature of chimney gases = 560° F.

Calculated results:

The carbon in the chimney gases being three and eight-tenths per cent. of their weight, the total weight of dry gases per pound of carbon burned is $100 \div 3.8 = 26.32$ pounds. Since the carbon burned is $80.55 - 2.0 = 78.55$ per cent. of the weight of the coal, the weight of the dry gases per pound of coal is $26.32 \times 78.55 \div 100 = 20.67$ pounds. Each pound of coal furnishes to the dry chimney gases 0.7825 pound C., 0.0108 N, and $\left(2.70 - \frac{4.50}{8}\right) \div 100 = 0.0214$ pound O; a total of 0.8177 or 0.82 pounds. This subtracted from 20.67 pounds leaves 19.85 pounds as the quantity of dry air (not including moisture) which enters the furnace per pound of coal, not counting the air required to burn the available hydrogen, that is, the hydrogen minus one-eight of the oxygen chemically combined in the coal.

Each pound of coal burned contained 0.045 pound of hydrogen, which requires $0.045 \times 8 = 0.36$ pound O for its combustion. Of this 0.027 pound is furnished by the coal itself, leaving 0.333 pound to come from the air. The quantity of air needed to supply this oxygen (air containing twenty-three per cent. by weight of O) is $0.333 \div 0.23 = 1.45$ pounds, which added to the 19.85 pounds already found gives 21.30 pounds as the quantity of dry air supplied to the furnace per pound of coal burned. The air carried in as vapor, 0.0071 pound for each pound of dry air, or $21.3 \times 0.0071 = 0.15$ pound for each pound of coal. Each pound of coal contained 0.029 pound of moisture, which was evaporated and carried into the chimney gases. The

0.045 pound of hydrogen per pound of coal when burned formed 0.045 × 9 = 0.405 pound of water.

From the analysis of the chimney gas it appears that 0.09 ÷ 3.80 = 2.37 per cent. of the carbon of the coal was burned to carbon monoxide instead of carbon dioxide.

We now have the data for calculating the various losses of heat, as follows, for each pound of coal burned:

	Heat units.	Per cent. of heat value of the coal.
21.3 pounds dry air × (560° − 60° F.) × sp. heat 0.238	2534.7	17.80
0.15 pound vapor in air × (560° − 60°) × sp. heat 0.48	36.0	0.25
0.029 pound moisture in coal heated from 60° to 212° F.	4.4	0.03
" " evaporated from and at 212°; 0.029 × 966	28.0	0.20
" " steam (heated from 212° F. to 560°) × 348 × 0.48	4.7	0.03
0.405 pounds water from H in coal × (560°—60°) × 0.48	97.2	0.68
0.0237 pound carbon burned to carbon monoxide, loss by incomplete combustion, 0.0237 × (14544 — 4451)	239.2	1.68
0.02 pound coal lost in ashes; 0.02 × 14544	290.9	2.04
Radiation and unaccounted for by difference	712.1	5.00
	3947.8	27.71
Utilized in making steam, equivalent evaporation 10.66 pounds from and at 212° per pound of coal	10295.7	72.29
	14243.0	100.00

The heat lost by radiation from the boiler and furnace is not easily determined directly, especially if the boiler is enclosed in brick work, or is protected by non-conducting covering. It is customary to estimate the heat lost by radiation by difference, that is, to charge radiation with all the heat lost which is not otherwise accounted for.

One method of determining the loss by radiation is to block off a portion of the grate surface and build a small fire in the remainder, and drive this fire with just enough draught to keep up the steam pressure and supply the heat lost by radiation without allowing any steam to be discharged, weighing the coal consumed for this purpose during a test of several hours duration.

Estimates of radiation by difference are apt to be greatly in error, as in this difference are accumulated all the errors of the analyses of the coal and of the gases. An average value of the heat lost by radiation from a boiler set in brick work is about four per cent.; when several boilers are in a battery and enclosed in a boiler house the loss by radiation may be very much less, since much of the heat radiated from the boiler is returned to it in the air supplied to the furnace, which is taken from the boiler room.

An important source of error in making a "heat balance," such as the one given above, especially when highly bituminous coal is used, may be due to the non-combustion of part of the hydrocarbon gases distilled from the cold immediately after firing, when the temperature of the furnace may be reduced below the point of ignition of the gases. Each pound of hydrogen which escapes burning is equivalent to a loss of heat in the furnace of 62500 B. T. U.

XVII.

The Determination of Sulphur in Steel and Cast Iron.

Of the various methods described for this purpose, the following three are selected as giving the best results in general practice:

(*a*). Bromine method.
(*b*). Aqua Regia method.
(*c*). Potassium permanganate method.

a. Bromine Method.

Dilute hydrochloric acid is allowed to act upon the steel or iron; the sulphur is expelled as hydrogen sulphide and is oxidized by the bromine to sulphuric acid.

This latter is precipitated by barium chloride as barium sulphate, filtered, washed and weighed as such, then calculated to sulphur.

The apparatus used is as follows:

In the flask *A*, capacity about 400 cc., is placed the steel or iron (five grams of steel or three grams of cast iron), and connection made with the absorption apparatus *D*.[1] In the latter,

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[1] All stoppers are of glass.

at *E*, is placed five drops of bromine and twenty-five cc. of hydrochloric acid (sp. gr. 1.18). Seventy-five cc. of hydrochloric acid (sp. gr. 1.12), is placed in the delivery funnel *B* and about ten cc. allowed to run into the flask. The action is quite often violent, and care must be exercised that small amounts of acid *only* be admitted at a time from *B* until all action of the acid upon the steel or iron ceases.

Heat is now gently applied, and contents of the flask brought to boiling; continue the boiling two or three minutes; remove the heat, connect the delivery tube *B* with a "Bennert drying apparatus," and connect the absorbent apparatus with an aspirator. Gradually aspirate about one liter of the air through the asparatus.

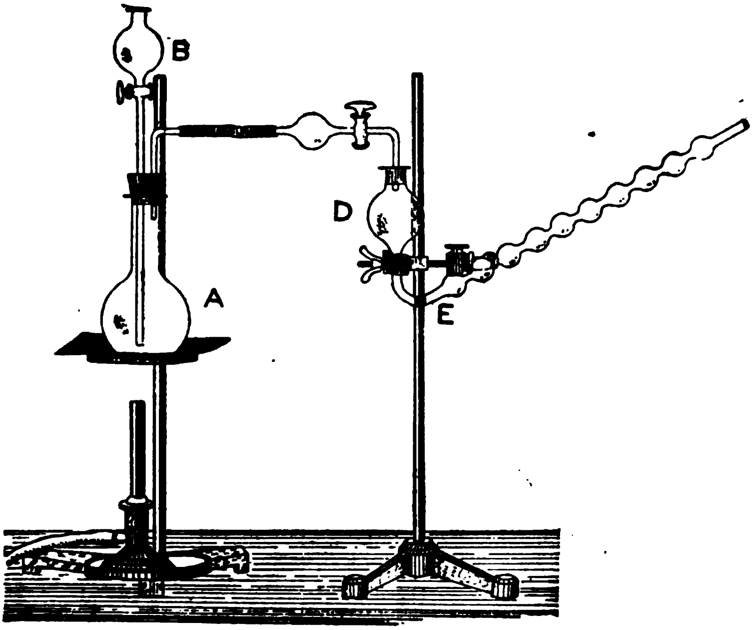

Fig. 42.

Between the aspirator and the absorbent apparatus there should be placed a wash bottle containing dilute ammonium hydroxide, (250 cc. strong ammonia to 600 cc. water), to absorb any fumes of bromine that may pass out of the absorbent apparatus during aspiration. Transfer the liquid in the absorbent tube to a No. 3 beaker, washing the tubes with water and add the washings to solution in beaker.

Bring to boil, expel any excess of bromine, add solution of barium chloride and set aside twelve hours. Filter upon two No. 3 ashless filters, wash with hot water, dry, ignite, and weigh as barium sulphate and calculate to sulphur.

b. Aqua Regia Method.

Five grams of the iron or steel (in fine turnings) are transferred to a No. 4 beaker and the latter covered with a watch-glass. Introduce into the beaker (in quantities not exceeding ten cc. each time) some nitric acid, until the iron or steel is dissolved. Warm gently and evaporate to dryness on an iron plate, adding some sodium carbonate previously, so that no sulphuric acid may be lost by vaporization.

Allow to cool, treat with hydrochloric acid, warm until solution of iron is complete and filter off the silica. Wash well and to the filtrate containing the washings add a few cc. of solution of barium chloride, and set aside twelve hours. Filter, wash with hot dilute hydrochloric acid, then with water thoroughly; dry, ignite, weigh as barium sulphate and calculate to sulphur.

This method is preferred where the iron or steel contains any metals (even in minute amounts) that are precipitated by hydrogen sulphide. Thus the presence of one-fourth per cent. of copper would render the bromine or permanganate process unreliable since hydrogen sulphide is generated, forming copper sulphide, and the resulting amount of sulphur would be too low.

In the "aqua regia method" the oxidation is performed at once upon the addition of the nitric acid, no hydrogen sulphide being formed.

c. The Potassium Permanganate Method.[1]

a is a flask holding 300 cc., with *pure* rubber stopper, through the latter passing a thistle tube with stop-cock for the delivery of the acid, as required. Fig. 43.

b is a flask with rubber stopper. The glass tubing must not reach below the neck of the flask. This flask should be large

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[1] *Trans. Amer. Inst. Mining Engineers*, 2, 224.

enough to hold the contents of the bottles in case back-suction should occur.

The bottles *c*, *d*, and *e* contain a solution of potassium per-

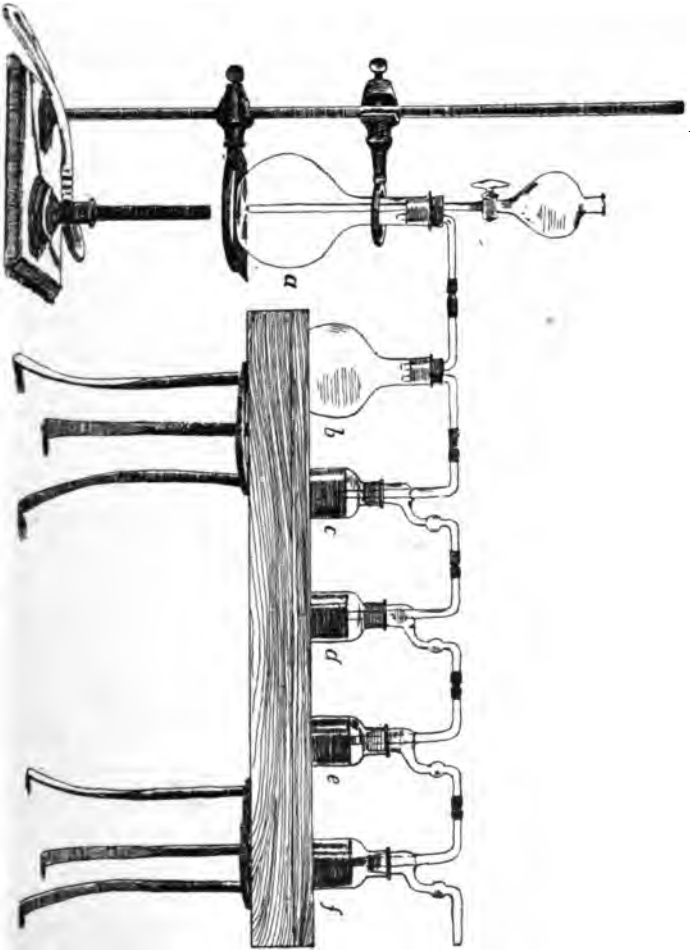

Fig. 43.

manganate, five grams potassium permanganate to 1000 cc. water, and are filled to the amount shown in the figure (about twenty-five cc. each).

f contains an ammoniacal solution of silver, and is used to test whether the hydrogen sulphide is all oxidized by the permanganate; if not oxidized, the solution in *f* becomes black from the silver sulphide formed.

The process is as follows:

Three grams of cast iron or six grams of steel are placed in flask *a* and hydrochloric acid (sp. gr. 1.12) gradually added until seventy-five cc. have been used. Warm contents of the flask, observing that the evolution of gas is not too rapid.

When the iron or steel is dissolved, bring the liquid to boiling, connect bottle *f* with an aspirator and slowly draw air through the apparatus ten minutes.

Transfer contents of bottles *c*, *d*, *e*, to a No. 3 beaker, dissolving any oxide of manganese that may have deposited in bottles with hydrochloric acid. Wash the bottles with hydrochloric acid, then with water, adding the washings to contents of the beaker.

The solution in the beaker is warmed and enough hydrochloric acid added that it becomes colorless or nearly so, and barium chloride added in sufficient quantity to precipitate the sulphuric acid. Allow to settle twelve hours. Filter upon two No. 2 ashless filters, wash with boiling water, dry, ignite, weigh as barium sulphate and calculate to sulphur.

Great care must be exercised in this process, that the potassium permanganate is free from sulphurous or sulphuric acid before use.

The iodine method for the determination of sulphur in pig iron and steel, as used by the chemists of the Duquesne Steel Works, is as follows:

Five grams pig iron or steel are weighed off into a dry 500 cc. flask, provided with a double perforated rubber stopper, with a long stem four ounce funnel tube with a stop-cock, and a delivery tube bent at right angles, on which a short piece of one-quarter inch rubber tubing is placed, making connection with a delivery tube, also bent at right angles reaching to the bottom of a one inch by ten inch test tube, suitably supported. About ten cc. of the ammoniacal solution of cadmium chloride is introduced into the test tube, which is diluted with cold water,

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[1] J. M. Camp: *Proceedings Engineers Society of Western Pa.*, 11, 251, 1895.

until the tube is about two-thirds full. Eighty cc. of dilute hydrochloric acid—one acid to two water—is poured into the funnel tube, a file marked on the bulb indicating this amount, which is allowed to run into the flask, the stop-cock is then closed, and a gentle heat applied, till the drillings are all in solution, and finally to boiling by raising the heat, until nothing but the steam escapes from the delivery tube.

The apparatus is then disconnected, and the delivery tube is placed in a No. 4 beaker in which the titrations are made, the contents of the test tube are then poured into the beaker, the test tube filled to the top twice with cold water, the sides of the tube rinsed down with about twenty-five cc. dilute hydrochloric acid and filled again with cold water. The total volume of the solution equaling about 400 cc., both acid and water being supplied from overhead aspirator bottles and suitable rubber connections with pinch cocks; the delivery tube is now rinsed off inside and out with dilute hydrochloric acid, and about five cc. starch solution added to the beaker.

Without waiting for complete solution of the cadmium sulphide, the iodine solution is run in from a burette, stirring gently, till a blue color is obtained, the solution is then stirred vigorously, keeping a blue color by fresh additions of the iodine solution, till the precipitate of cadmium sulphide is all dissolved, and the proper permanent blue color is obtained. The amount of iodine solution used in cc. is hundredths per cent. sulphur.

Iodine solution is made by weighing off into a dry 500 cc. flask about thirty-five grams potassium iodide, and sixteen grams iodine, fifty cc. water added and shaken and diluted cautiously until all are in solution, and finally diluted to 3500 cc. This is standardized with steels of known sulphur contents, so that one cc. equals 0.0005 grams sulphur.

Cadmium chloride solution is made by dissolving 100 grams cadmium chloride in one liter water, adding 500 cc. strong ammonia, and filtering into an eight liter bottle; two liters of water are now added, and the bottle filled to the eight liter mark with strong ammonia.

Starch solution is made by adding to one-half gallon boiling water, in a gallon flask, about twenty-five grams pure wheat starch, previously stirred up into a thin paste with cold water; this is boiled ten minutes and about twenty-five grams pure granulated zinc chloride dissolved in water added, and the solution diluted with cold water to the gallon mark. The solution is mixed and set aside over night to settle, the clear solution is decanted into a glass stoppered bottle for use.

This solution will keep indefinitely.

References: "Volumetric Method of Elliott," *Chem. News*, 23, 61.

"Wiborgh's Colorimetric Method," *J. Anal. Chem.*, 6, 301.

"Sulphur Determinations in Iron and Steel," by different methods. By L. S. Clymer, *J. Anal. Chem.*, 4, 318.

"Cadmium Chloride as an Absorbent of Hydrogen Sulphide"(Sulphur in Iron and Steel). By Frank L. Crobaugh, *J. Anal. Chem.*, 7, 280.

"The Reduction of Barium Sulphate to Sulphide on Ignition with Filter Paper." By C. W. Marsh, *J. Anal. Chem.*, 3, 2.

"Duplicate Determinations of Sulphur in Iron and Steel Should Agree within 0.005 Per Cent." By C. B. Dudley, *J. Am. Chem. Soc.*, 15, 514.

"The Determination of Sulphur in Iron and Steel. By L. Archbutt, F.I.C., *J. Soc. Chem. Industry*, 4, 75.

XVIII.

Determination of Silicon in Iron and Steel.

Five grams of steel or three grams of pig iron in fine borings, are transferred to a No. 3 beaker[1] and fifty cc. of dilute sulphuric acid added. When the action of the acid ceases and the iron is dissolved, twenty-five cc. nitric acid (sp. gr. 1.20) is cautiously added until effervescence ceases.

Apply heat and evaporate until white fumes of sulphur trioxide appear; allow to cool; add strong hydrochloric acid until the residue is thoroughly saturated with it, then add seventy-cc. boiling water.

Filter, wash with dilute hydrochloric acid, then with hot water, dry, ignite, weigh as silicon dioxide and calculate to silicon.

This method must be used in the determination of silicon in pig iron, but in wrought iron and steel the insoluble residue in the determination of phosphorus may be used for the silicon, if desired.

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[1] Porcelain beakers are to be preferred to glass beakers for this determination.

In all determinations of this element, the ignited and weighed silicon dioxide must be white in color and a fine non-coherent powder.

References: "Irregular Distribution of Silicon in Pig Iron." By J. W. Thomas, *J. Anal. Chem.*, **2**, 148.

"Silicon in Pig Iron." By Clemens Jones, *J. Anal. Chem.*, **3**, 121.

"The Influence of Silicon on the Determination of Phosphorus in Iron." By Thomas M. Drown, *J. Anal. Chem.*, **3**, 288.

"Notes on Silicon in Foundry Pig Iron. By David H. Brown, *J. Anal. Chem.*, **6**, 452–467.

XIX.

The Determination of Carbon in Iron and Steel.

The determination of carbon in iron and steel has probably received more attention in later years from chemists than any other subject in analytical chemistry.

To secure a method at once complete and rapid whereby carbon varying in amounts from four per cent to 0.001 per cent. in different irons and steels could be accurately determined has been a *desideratum*.

Processes that are satisfactory for special grades of irons or steels rarely can be relied upon in general practice.

So important has this subject become to the metallurgical world that committees acting in union from Sweden, England, and America[1] have been appointed to determine not only the best methods of iron and steel analysis, but also to analyze standard samples of iron and steel, compare the results, and select methods which should be uniform for the different countries.

The determination of carbon, as made upon the standard samples, are thus reported:

Standard.	No. 1. Per cent.	No. 2. Per cent.	No. 3. Per cent.	No. 4. Per cent.
English committee······	1.414	0.816	0.476	0.151
Swedish committee······	1.450	0.840	0.500	0.170
American committee····	1.440	0.807	0.452	0.160

The English and Swedish committees have not yet selected

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[1] *J. Am. Chem. Soc.*, 15, 449.

the method to be adopted as standard in carbon determinations, but the American committee have rendered their report suggesting certain modifications in the use of solvents for the iron and the separation of the total carbon.

The use of the double chloride of copper and potassium, as a solvent for iron, is recommended in place of the double salt of chloride of copper and ammonium, owing to the great difficulty in obtaining the latter salt free from pyridin and other tarry products.

Of the many methods used to obtain the amount of carbon from the iron, the following few are selected to indicate not only the variety of the processes, but a gradual improvement by combination of different methods :

Berzelius[1] first suggested that the iron or steel be finely pulverized and then ignited in a current of oxygen and the resulting carbon dioxide weighed.

Regnault[2] made use of combustion of the powdered iron with chromate of lead and chlorate of potash and the amount of carbon dioxide weighed.

Berzelius, however, in 1840, separated the carbon from the iron by dissolving the latter in copper chloride and igniting the carbon in oxygen.[3]

From this period, the methods for total carbon can be included in two general classes :

First class.—Combustion of the carbon in the powdered iron directly.

Second class.—Separation of the carbon from the iron by chemical means and combustion of the carbon.

Deville and Wöhler[4] describe processes by which the iron can be separated from the carbon, by volatilization of the iron with chlorine or hydrochloric acid gas, and combustion of the remaining carbon.

With the exception of a method described by Gmelin[5] by which the powdered iron is treated directly with chromium tri-

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[1] *Ann. phys. chem.*, 1838.

[2] *Ann. chim. phys.*, 1839, 107.

[3] *J. prakt. Chem.*, *1840*, 247.

[4] *Ztschr. anal. Chem.*, 8, 401.

[5] *Oesitericher Zeitschrift für Berg und Hüttenwesen*, *1883*, 392.

oxide and sulphuric acid and the carbon oxidized to carbon dioxide, the methods of the first class, above given, are no longer used.

Second Class.—These methods give better results in general practice, and nearly all the advances and improvements have been made in this direction.

Ullgren[1] dissolved the iron with solution of copper sulphate and oxidized the carbon to carbon dioxide by heating with chromium trioxide and sulphuric acid.

Eggertz[2] dissolved the iron with bromine or iodine, and the separated carbon was ignited with chromate of potash.

Langley[3] modified Ullgren's method by ignition of the carbon in oxygen after solution of the iron by copper sulphate.

Richter dissolved the iron with chloride of copper and potassium and burned the carbon in oxygen.

Weyl and Binks dissolved the iron in dilute hydrochloric acid passing an electric current at the same time and ignited the carbon in oxygen.[4]

Parry dissolved the iron in solution of copper sulphate, the carbon burned, mixed with copper oxide, in vacuo, and the volume of carbon dioxide measured.[5]

Eggertz's method for combined carbon[6], in which the iron was dissolved in nitric acid and the amount of carbon (combined) determined by color of the solution formed.

McCreath and Pearse dissolved the iron with chloride of copper and ammonium and ignited the carbon in oxygen.[7]

Boussingault[8] decomposed with mercuric chloride and oxidized the carbon to carbon dioxide.

Wiborgh[9] dissolved the iron with solution of copper sulphate, oxidized the carbon by heating with chromium trioxide and sulphuric acid, and measured the volume of carbon dioxide formed.

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[1] *Ann. de Chem. u Phar.*, 124, 59.
[2] *Dingler's Polytechnishes Journal*, 170, 350.
[3] *American Chemist*, 6, 265.
[4] *Ann. phys. Chem.*, 114, 507.
[5] *Chem. News*, 25, 301.
[6] *Chem, News*, 7, 254.
[7] *Engineering and Mining Journal*, 21, 151.
[8] *Dingler's Polytech. J.*, 197, 25.
[9] *Dingler's Polytech. J.*, 265, 502.

Experience has shown that the methods of Ullgren, Langley, Richter, and Wiborgh give the best results for the total amount of carbon in iron, and that the Eggertz method for combined carbon in steel can be relied upon as the best for the purpose.

The determination of total carbon, as made in my laboratory, is either by the Ullgren or Langley methods, somewhat modified. The Ullgren method is thus performed: Six grams of the iron, in fine turnings, are transferred to a No. 3 beaker and 100 cc. of a solution of copper sulphate[1] (1 to 5) added, the solution being first rendered neutral by a few drops of a very dilute solution of potassium hydroxide. Digest at a gentle heat until all the iron is dissolved (no smell of hydrocarbon given off), add 100 cc. cuprous chloride solution (1 to 2) and seventy-five cc. hydrochloric acid (specific gravity 1.2) and warm until the metallic copper is dissolved. Filter upon an asbestos filter, washing first with dilute hydrochloric acid, and finally with water until no reaction for hydrochloric acid is obtainable with a drop of silver nitrate solution. Transfer the asbestos filter containing the carbon to the flask *A*, Fig. 44, using not over twenty-five cc. water in the operation. Add ten grams chromium trioxide, and in the delivery flask place fifty cc. concentrated sulphuric acid, and connect the flask with the system of U-tubes.

B contains water sufficient to cover the neck of the U-tube, and is made slightly acid with sulphuric acid.

C and *D* contain granulated calcium chloride free from lime.

E and *F* contain soda lime, medium granulated, and are carefully weighed before use.

G contains granulated calcium chloride. Allow the sulphuric acid in the delivery tube to enter flask *A* and close the stop-cock. Warm the contents of the flask gradually to boiling, and when no more gas passes through *B* open the side stopcock of flask *A* and connect with the Trauber drying apparatus. The aspirator is connected with *G* and the air is slowly aspirated through the entire apparatus. Continue this until about five liters of air have been aspirated.

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[1] Copper chloride and hydrochloric acid can be substituted as recommended by American Committee on Standard Methods.

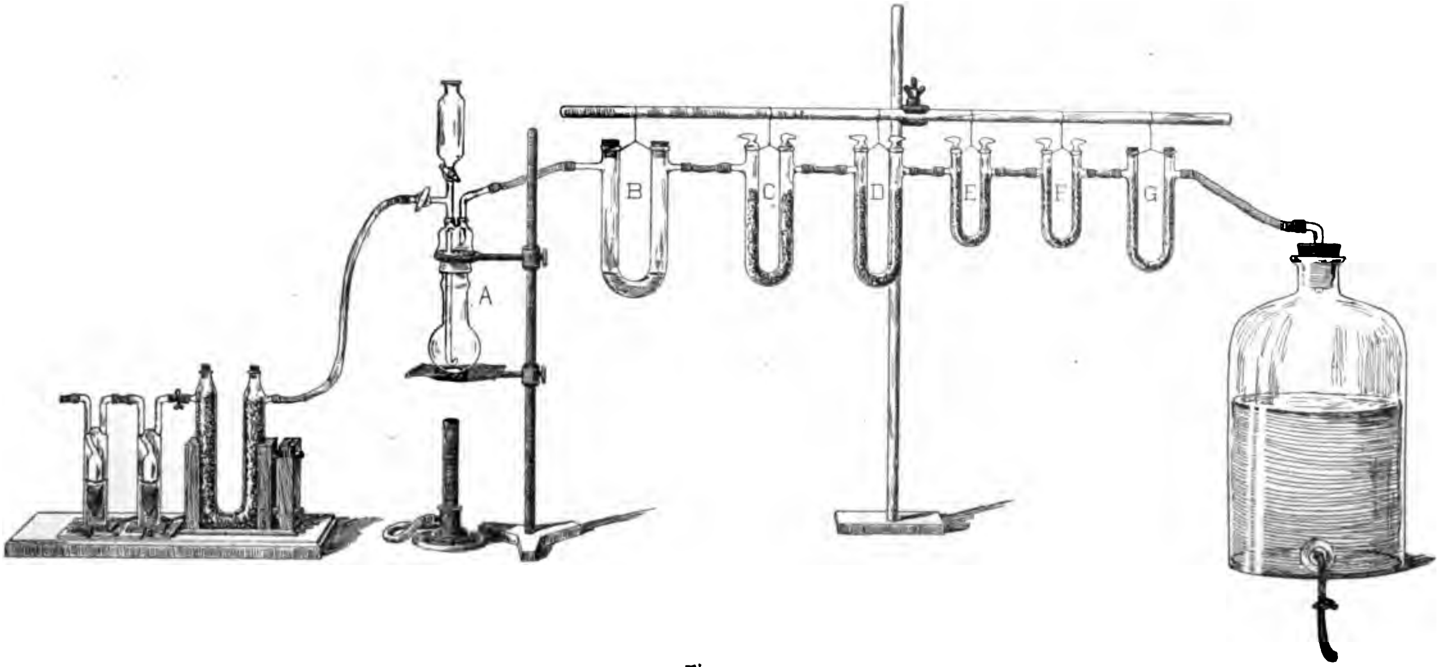

Fig. 44.

After twenty minutes weigh tubes *E* and *F*; the increase of weight represents the carbon dioxide produced by the oxidation of the carbon.

Thus, six grams of cast iron taken:

Tubes *E* and *F* + CO_2	65.700
Tubes *E* and *F*	65.002
CO_2	0.698

$$CO_2 : C :: 0.698 : x = 0.1904.$$

$$\frac{0.1904 \times 100}{6} = 3.18 \text{ per cent. carbon.}$$

The carbon in cast iron being generally a mixture of combined and graphitic carbon, it is essential to determine the graphitic carbon, and this amount being subtracted from the total carbon gives the combined carbon. In steels where the carbon is all combined the color test of Eggertz suffices. The graphite is thus determined:

Add fifty cc. hydrochloric acid (specific gravity 1.1) to six grams of cast iron or ten grams of steel in a No. 3 beaker; warm gently until the iron is all dissolved, bring to boiling temperature for five minutes, allow the graphite to settle, and decant the supernatant liquid upon an asbestos filter; wash by decantation four times with hot water and treat residue in beaker with twenty-five cc. solution of potassium hydroxide (sp. gr. 1.12) and boil. Transfer to the asbestos filter, wash thoroughly with boiling water, then with alcohol and ether, and transfer the asbestos filter to the flask *A*, Fig. 44, and oxidize the carbon to carbon dioxide with chromium trioxide and sulphuric acid, as in the process previously given for total carbon.

Thus, six grams of iron taken:

Tubes *E* and *F* + CO_2	= 66.053 grams.
Tubes *E* and *F*	= 65.621 "
CO_2	= 0.432 "

$C = 0.1178$ gram.

Graphitic carbon	= 1.96 per cent.
Combined carbon	= 1.22 "
Total carbon	3.18 "

Method of Langley Modified.

In this process the sample is treated in the same manner for solution of the iron as described for total carbon in the Ullgren method. After the carbon has been thoroughly washed upon the asbestos filter, it is dried and transferred to a porcelain boat which is placed inside of a combustion tube in the furnace *C*, Fig. 45.

The tube *D* connected with the combustion tube contains granulated calcium chloride, and *E* and *F* soda lime; another tube *G* containing calcium chloride (not shown in the figure), is also used. Oxygen under pressure in the tank *A* is allowed to pass slowly through the Trauber drying apparatus, which removes all moisture and carbon dioxide, into the combustion tube and through the tubes *D*, *E*, *F* and *G*.

Heat is gradually turned on in the furnace and increased until the carbon is completely burned to carbon dioxide. Turn off the heat, disconnect the oxygen tank and slowly aspirate air through the apparatus by means of an aspirator.

After cooling thirty minutes weigh the tubes *E* and *F* and calculate the result as given previously.

It will be noticed that no Liebig's potash bulbs are used. I have obtained better results by the use of soda lime in U-tubes than by the potash bulbs, and in general practice they will be found much more convenient and less liable to variation in weight.

The use of the double chloride of copper and ammonium as a solvent for the iron has been quite general in this country. The American Committee on Standard Methods of Iron Analyses found that, contrary to the usual practice, this solvent must not be neutral, but strongly acid with from five to ten per cent. of its volume of strong hydrochloric acid.

T. M. Drown, in his report to the committee, describes his process as follows: "Three grams of the steel were treated with 200 cc. of a solution of copper potassium chloride (300 grams to the liter) and fifteen cc. of hydrochloric acid (sp. gr. 1.2). After complete solution of the iron the carbon was filtered off on an asbestos lined platinum boat, thoroughly washed with hy-

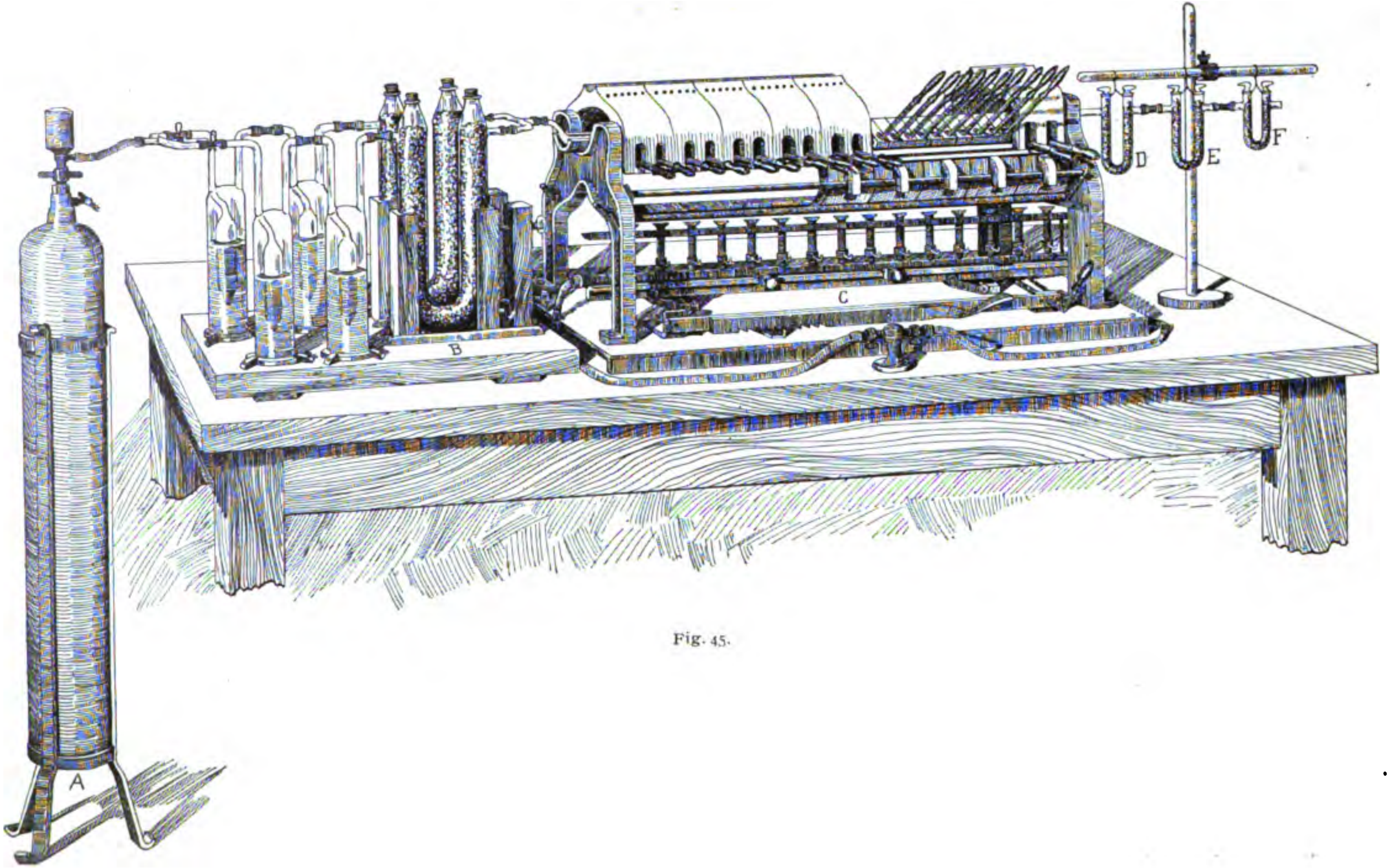

Fig. 45.

drochloric acid, and then with water until the washings gave no reaction with silver nitrate. After drying the boat was put into a porcelain tube and the carbon burned in a current of oxygen." This is a modification of Richter's process.

There does not appear to be much choice in the method of the combustion of carbon. Some chemists prefer oxidation with chromium trioxide and sulphuric acid, and others ignition in a current of oxygen gas.

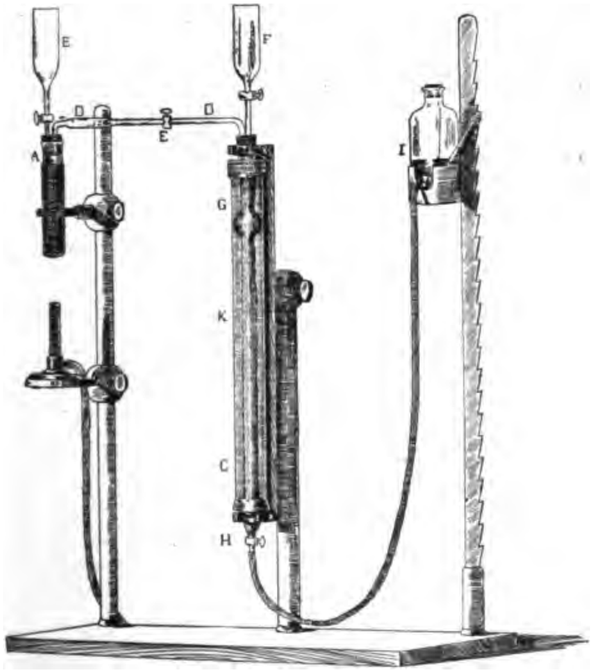

Fig. 46.

For rapidity of execution and simplicity of apparatus (Fig. 44), I prefer the former.

Wiborg's method,[1] in which the carbon dioxide is measured instead of being weighed, consists as follows: The apparatus required is shown in Fig. 46. A test tube *A*, 140 mm. long by twenty mm. internal diameter, is surrounded by a cage of brass

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[1] *J. Soc. Chem. Industry*, 6, 748.

wire gauge, and fitted with a caoutchouc cork with two perforations. Through one perforation passes the narrow end of the stop-cock funnel *B*, which should project for about fifteen to twenty mm. beneath the cork; through the other, but not projecting beneath the stopper, passes the connecting tube *D*. This latter tube consists of two portions, united by India rubber tubing; the part more remote from *A* and carrying the stop-cock *E* is bent to pass through one of the perforations of another caoutchouc stopper in the graduated tube *C*, the other perforation serving to connect the latter with a stop-cock funnel *F*.

The tube *C* should for the distance of seventy mm. downwards have an internal diameter of sixteen mm; it should then be widened to a bulb *G*, of about twenty-five centimeters capacity, and be finally reduced for the remaining 200 mm. to about nine mm., this narrow portion being graduated into divisions of one-tenth, or preferably, one-twentieth of a cubic centimeter, denoting in each case the capacity of the whole of that portion of the tube above the respective graduations. Beneath this tube is the stop-cock *H*, communicating by flexible tubing with the movable water reservoir *I*. The test tube *A* is warmed by a gas or spirit lamp, and the whole apparatus should be mounted on a suitable stand. The measuring tube is surrounded by a water jacket *K* to preserve an even temperature.

To conduct an analysis two-tenths gram of finely divided wrought iron or steel or one-tenth gram of cast iron is introduced carefully into the test tube *A*, taking care that none of the filings adhere to its sides. Four cc. of a saturated solution of pure copper sulphate are then introduced and allowed to act, with frequent stirring, during ten minutes, unless an appreciable smell of hydrocarbon is observed, when the action must be suspended after three or four minutes. One and two-tenths grams of crystallized chromic acid are added to the solution. Meanwhile the tube *C* must have been filled with water by raising the reservoir *I* until the liquid has risen above the bulb tube *G*, the remaining space up to the cock being filled by water introduced through *F*. The test tube is now corked and connected with the burette *C*.

Eight cc. of sulphuric acid (sp. gr. 1.7) are introduced drop by drop into *A* through *B*, the cock of the latter is closed, that marked *E* opened, and the liquid in the test tube gradually raised to boiling, the pressure having been diminished by previously lowering the water reservoir *I*. After ten minutes' boiling, during which the reservoir has been still further lowered, if necessary, to maintain the diminished pressure, the tube is cooled somewhat, and, together with the connecting tube *D*, is carefully filled with water introduced through *B*. The cock *E* is then closed and the total volume of air and carbon dioxide read off after leveling with the reservoir.

I is then once more lowered and the cock *H* closed in order to draw in a quantity of a ten per cent. potassium hydroxide solution through *F*. After the carbon dioxide has been completely absorbed, *H* is reopened, the liquid leveled again and a reading of the residual air is taken.

The difference between the two readings will be the volume of carbon dioxide evolved from the carbon in the iron.

Evidently if two-tenths gram of substance were used, each cc. of carbon dioxide will correspond to 0.253 per cent. of carbon, and the factor 0.253 multiplied by the number of cubic centimeters of gas should give a direct reading of the percentage of carbon.

But this is not quite correct, since a certain quantity of carbon dioxide (to be found by experiment) is absorbed by the water in the tube. By treating pure anhydrous sodium carbonate in the apparatus instead of iron and comparing the actual with the theoretical yield of carbon dioxide, the factor may be corrected.

Thus the true factor was found to be 0.28, and this was universally correct for cast irons; but for wrought irons or steels, which contain less carbon, it should be 0.29.

When one-tenth gram of iron is used the factor must of course be doubled.

Where the temperature of the operation differs much from the normal eighteen degrees, correction must be made by multiplying or dividing by $(1 + 0.00367 \times t)$, where t is the variation in temperature, according as the solution is cooler or warmer than the normal.

This process is expeditious, and a very delicate measurement of the carbon dioxide can be obtained, thus:

One-twentieth cc. of carbon dioxide from two-tenths gram of iron represents 0.014 per cent. of carbon, but weighs only 0.0001 gram.

G. Lunge[1] gives this process the preference where small quantities of carbon are to be determined in cast irons.

Determination of Combined Carbon in Steel. Eggertz' Method.

This method depends upon the color given to nitric acid (sp. gr. 1.2) when steel is dissolved therein; the carbon present producing a light brown or dark brown coloration to the liquid in proportion as the carbon is in small or large amounts. The apparatus, Fig. 47, is well arranged for this test. It consists of a series of graduated tubes, of glass, each 27.5 centimeters long, fifteen mm. in diameter, and graduated to hold thirty cc. divided by one-fifth cc. The back plate of the apparatus is of white porcelain, 25.5 centimeters wide, twenty-seven centimeters high, and three mm. thick, and I have found it much better than the various cameras to obtain correct compari-

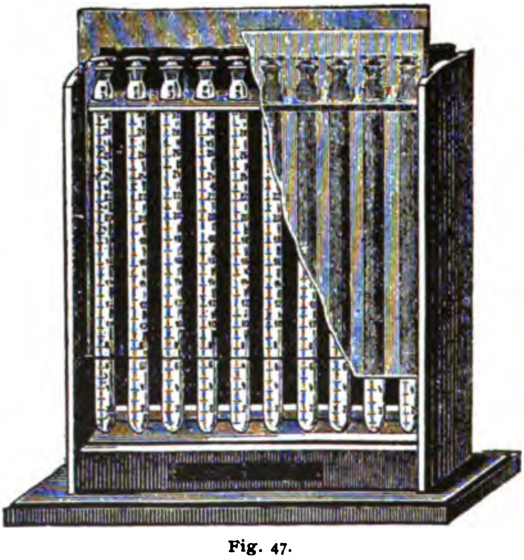

Fig. 47.

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[1] *Stahl und Eisen*, 13. 655.

sons of colors of solutions in the different tubes. Three standard steels are required, one containing one per cent. combined carbon, for tool steels, etc., one containing four-tenths per cent. carbon, for tires, rails, etc., and two-tenths per cent. carbon, for soft steels; these percentages of carbon having been very accurately determined by combustion.

The process is as follows: Two-tenth gram of the standard steel is transferred to one of the graduated tubes, four cc. of nitric acid (sp. gr. 1.20) added, and the tube placed in cold water to prevent energetic action of the acid. After a few minutes interval the tube is placed in warm water, and the latter gradually raised to the boiling-point and maintained at that temperature about twenty minutes. The sample of steel, in which the amount of carbon is unknown, is treated in a similar manner, using the same amount of steel and acid.

Suppose the standard steel contains 0.84 per cent. of carbon, the solution in the tube is diluted with water to 16.8 cc. Each cubic centimeter therefore contains 0.0001 gram of carbon. Suppose that upon dilution of the test sample solution to fourteen cc., and placing the two tubes side by side in the frame (Fig. 47), that the test sample is somewhat stronger in color than the standard sample; upon diluting it, however, to fifteen cc. it is slightly lighter in color. This would indicate that the unknown or test sample contains more than 0.70 per cent. ($0.1 \times \frac{14}{2}$) of carbon, but less than 0.75 per cent. ($0.1 \times \frac{15}{2}$). The steel can be thus assumed to contain 0.73 per cent. carbon.

The use of Eggertz' color test for combined carbon requires that steels should have been subjected to a similar physical treatment to which the standard steels had been subjected in order to secure accurate results. A steel shows less carbon, by color, when hardened than when unhardened, and less unannealed than when annealed. Several modifications of the process have been submitted by various chemists, but they offer no special advantages. Stead[1] renders the nitric acid solution of the steel alkaline with sodium hydroxide, which dissolves the carbon, producing a solution about two and a half times stronger in color than the solution in nitric acid. The precipitated iron

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[1] *Chem News*, 47, 285.

oxide is filtered off, and a measured quantity of the colored filtrate is transferred to a Stead's chromometer and the color compared with a standard steel under similar conditions. Except where the carbon is present in minute quantity only is this process of any advantage over the Eggertz method.

Carbon Compounds of Iron.

Microscopical examinations of iron have led to remarkable developments of our knowledge of its structure. Recent investigations by Osmond, Martens, Arnold and others have shown that eminently practical results are to be obtained from this microscopical examination.

These microscopical examinations indicate that structure of iron depends upon a number of partially identified compounds which have been given the names of pearlite, cementite, martensite, etc., and which are all compounds of carbon and iron.

Marten's and Osmond's latest investigations, as well as those of Arnold, have demonstrated that the presence or absence of one or more of these compounds determines and identifies the qualities and properties of different kinds of iron and also determines the methods of manufacture and heat treatment to which they were subjected.

Messrs. Abel, Mueller, and Leduber showed some years ago that carbon in unhardened steel exists chiefly as the definite carbide, Fe_3C; but microscopical investigations have further proven the coexistence of many other carbides, especially after heat treatment. Professor Arnold claims to have proven the existence of:

(*a*) Crystals of pure iron which remain bright upon etching.

(*b*) Crystals of slightly impure iron which become pale brown on etching, probably owing to the presence of a small quantity of an intermediate carbide of hypothetical formula $Fe_{10}C$.

(*c*) Normal carbide of iron, Fe_3C, which exists in three distinct modifications; each one conferring upon the iron in which it is found particular mechanical properties:

(1) Emulsified carbide present in an excessively fine state of division in tempered steels.

(2) Diffused carbide of iron occurring in normal irons in the forms of small ill-defined striæ and granules.

(5) Crystallized Fe_3C occurring as well defined laminæ in annealed and in some normal irons.

(*d*) Subcarbide of iron, a compound of great hardness existing in hardened and tempered irons and possessing formula $Fe_{24}C$. This substance is decomposed by the most dilute acids, and at 400° C. it is decomposed into Fe_3C and free iron with evolution of heat. One of the most remarkable properties of this compound is its capacity for permanent magnetism.

(*e*) Graphite or temper carbon.

Chemists have heretofore identified only graphitic carbon, combined carbon, and carbide of iron. These alone are not sufficient to identify iron, and what must be done is to devise accurate methods for determining all of the above enumerated substances by chemical analysis.

Professor Arnold says: "The existence of $Fe_{24}C$ is proved by the fact that iron containing 0.89 per cent. carbon presents several co-relative critical points when examined by different methods of observation: (1) Well marked saturation points in micro-structure of normal annealed and hardened steels. (2) A sharp maximum in a curve, the coordinates of which are heat evolved or absorbed at Ar 1 (point of recalescence) and carbon percentage. (3) A point in the compression curve of hardened steels at which molecular flow absolutely ceases. (4) A sharp maximum in a curve, the coordinates of which are carbon percentage and permanent magnetism in hardened steels."

The famous French micrographist, F. Osmond, defines and describes five distinct carbon compounds which can only be found and identified by the microscope, as existing in iron subjected to heat treatment.

(1) The first he calls, with Howe, ferrite, because it is almost pure iron; it at first retains a dull polish (poli spéculaire) when relief polished; after continued polishing, especially with precipitated chalk and water, it becomes more granular as it is less massive, but when forming large masses it finally shows as polyhedral crystals. Etch polishing with tincture of iodine produces no coloration.

(2) The second is called cementite, which is distinguished by its hardness (felspar, No. 6, Mohr's scale). This hardness, which is greater than that of all other carbon compounds, permits its identification even when polishing with emery paper, provided it is not so imbedded in softer particles, that the microscope is no longer able to identify it, and chemical analysis alone is able to prove its presence. This substance corresponds to that imagined by Karsten and Caron, and isolated by Dr. F. C. G. Mueller, Sir Fred. Abel, and Professor Ledebur as carbide, and of the probable formula Fe_3C, and which Howe also calls cementite. Osmond believes that cementite of iron of cementation (de l'acier poule) can now be identified with the hard component of cast and forged steels.

(3) The third compound is called sorbite, after Dr. Sorby, which was first described as "pearly constituent," (Howe called it perlite), which could be identified under a magnification of 800 diameters, as a substance with the sheen of mother-of-pearl. It is possible, with oblique light, to separate this in bands of alternating hard and soft flakes.

Osmond questions the accuracy of the conclusion generally held that this is Fe_3C, because he points out that etch polishing gradually changes the color from yellow to brown and then from purple to blue, and at a certain period there is great difference between the colors in adjacent "islets" (ilots).

The uncolored flakes (lamelles) may appear elevated or depressed. With tincture of iodine similar results are obtained. It must be remembered that neither ferrite nor cementite take such colors under similar conditions even when extract of liquorice root or tincture of iodine is used.

He can offer no suggestion in regard to the chemical composition of "sorbite."

(4) A fourth compound always found after quenching iron, which is already well known, is "martensite," named after Professor A. Martens, the famous micrographer of Berlin.

When iron with 0.45 per cent. carbon is heated to 825° C., and then at 720° C. quenched in a freezing mixture of 20° C., relief polishing produces no effect; but etch polishing shows the structure. Groups of needles (fascicles) or groups of rectilinear parallel fibers, which are separated or not by a scarry or vermiform filling, and are shown in very slight depths. Three groups of fibers, parallel to the sides of a triangle, are often seen in one spot, as crystalline bodies of the cubical system. Etch polishing does not always color martensite, and then only takes a light yellow sheen. However, when applying tincture of iodine it takes a yellow, brown, or black color, according to the percentage of carbon present. Because of the non-uniformity of color it is not quite certain whether martensite can be considered a fundamental compound. It does, however, retain its forms even in the quenched parts, in the softest as well as in the hardest iron, with the single difference that the fascicles (needles) are sometimes longer, sometimes more varied, in accordance whether the iron is more or less carbonized. The shapes are characteristic and permit the determination of differences in hardness. Martensite is not positively a definite compound of iron and carbon ; it represents rather the crystalline arrangement of an allotrapic modification of iron under the influence of carbon.

(5) A fifth well defined fundamental compound found in medium iron quenched while undergoing structural changes (into its allotropic modifications according to Osmond), is named troostite, after the famous French metallurgist Troost. When iron with 0.45 per cent. carbon is heated to 825° C. and then quenched at 690° C., is relief polished, nodules in relief, depressed tatters or tongues (lambeaux), and between the two intercalations of varying breadth and medium hardness are developed. Etch polishing proves that the hard nodules are martensite, and the soft tatters or tongues are ferrite. The intercalated bands show temper colorations, but they harden less rapidly than sorbite under identical conditions, and these colors produce an irregular marbleized appearance ; they are almost amorphous, slightly granular and warty. Tincture of iodine in first and second application produces quite similar effects in this fifth fundamental compound, troostite.

It is noticed that it is a transitory form between soft iron and hardened steel. But troostite is identified by the microscope alone, just like the sorbite ; its composite character is still to be determined. The systematic microscopic examination consists briefly in the application of three methods:

(1) Relief polishing, (2), etch polishing, and (3) etching with tincture of iodine.

In relief polishing it is sometimes advisable to use precipitated chalk as well as rouge, to preserve the ferrite.

In etch polishing with precipitated chalk, the fundamental compounds, with the exception of martensite, are divided into two groups:

(*a*) Not colored: ferrite, cementite, or martensite.

(*b*) Colored: martensite, troostite, or sorbite.

Martensite takes only a yellowish color and is distinguishable by its crystalline form. A novice might take martensite for perlite, especially by oblique light, for both have irridescent sheen, and its structural elements may be of equal dimensions; but they are easily distinguished, as the needles of martensite are straight and crossed, while those of perlite are curved and never cross each other.

Ferrite and cementite are distinguished by their great differences in hardness; the former is low, the latter is high. Troostite takes less color and more slowly than sorbite, but the true distinctive mark is that troostite accompanies martensite, while sorbite goes with cementite in perlite.

By etching with tincture of iodine two groups can be distinguished, *viz.*:

(*a*) Uncolored: ferrite and cementite.

(*b*) Colored: sorbite, troostite and martensite.

In group (*b*) the three compounds vary in color, in kind and depth in proportion to the percentage of carbon and of the quantity of tincture of iodine used.

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XX.

Determination of Phosphorus in Cast Iron and Steel.

The molybdate method, as described by Troilius,[1] gives uniform and satisfactory results. It is as follows: Five grams of drillings are dissolved in a No. 4 Griffin's beaker, in nitric acid (sp. gr. 1.20), using about fifty cc. of the acid. The solution is then evaporated with excess of strong hydrochloric acid by rapid boiling on a large iron plate by one of Fletcher's solid flame burners.

The plate is so heated that the heat gradually decreases from the centre towards the edges. The hottest part ought to be rather above than below 300° C. The evaporation is continued on the hottest part of the plate until signs of spattering are noticed. The beaker, or beakers, are then moved to a less hot part of the plate. When the tendency to spatter has ceased the beakers are moved back to the hottest part of the plate for at least half an hour. This heating is necessary in order to completely oxidize and decompose the last traces of iron phosphide, which would otherwise remain insoluble with the silica. The presence of hydrochloric acid lessens the tendency to spatter, which is always less in high carbon steels than in low carbon steels.

The beakers are now slowly cooled and strong hydrochloric acid added in excess. This acid is at once brought to a boil, which effects a solution of the residue, and the boiling is continued until only a small bulk remains.

This boiling serves two purposes:

1. To convert any pyrophosphoric acid ($H_4P_2O_7$), which may have been formed by the strong heating into orthophosphoric acid (H_3PO_4).

2. To concentrate the solution and remove the excess of hydrochloric acid which would otherwise interfere with the precipitation of phosphoric acid by means of molybdic acid.

Hot water is added and the insoluble residue filtered off and thoroughly washed with dilute hydrochloric acid, and afterwards with hot water.

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[1] "Notes on the Chemistry of Iron," by Magnus Troilius, E. M.

The phosphoric acid in the filtrate is precipitated as the yellow phospho-molybdate of ammonia.

For this precipitation is used a solution of about one part by weight of molybdic acid in four weights of ammonia (0.96 sp. gr.), and fifteen parts of nitric acid (1.20 sp. gr.). The molybdic acid is first dissolved in the ammonia, and this solution slowly poured *into* the nitric acid, which must be shaken constantly in order to prevent the separation of molybdic acid, which redissolves with difficulty. After a few days' standing the solution may be siphoned off clear. Fifty to one hundred cc. of this solution are used for each phosphorus determination.

To precipitate the phosphoric acid in the filtrate from the insoluble residue (silica, etc.) sufficient ammonia is added to nearly neutralize the solution. The fifty cc. of molybdic acid solution are then added and the solution well stirred. If the yellow precipitate is slow in coming down, a little more ammonia may be added. If too much ammonia is added, a little strong nitric acid must be introduced to redissolve the iron precipitate. As a rule the yellow precipitate comes down very quickly. By neutralizing the solution *before* adding the molybdic acid, as described, the yellow precipitate becomes granular and easy to filter. When precipitated in any other way it has a tendency to pass through and creep over the edges of the filter.

The yellow precipitate is allowed to settle over night at about 40° C, or during a few hours at 80° C.

After settling the clear supernatant liquid is siphoned off and the precipitate washed with copious quantities of molybdic acid solution diluted with an equal volume of water. About 300 cc. of washing are not too much to insure the complete removal of the last traces of iron. The yellow precipitate is then treated on the filter with six cc. hot ammonia (0.96 sp. gr.) and the filtrate allowed to run back into the beaker in which the precipitation was made. When all is dissolved the ammoniacal solution is thrown on the same filter again, but now allowed to run into a 100 cc. beaker. The filter is then washed well with small portions of cold water, so that the bulk of the ammoniacal solution will not exceed forty cc. This is now made faintly acid with hydrochloric acid, then alkaline with a few drops of ammonia, enough to dissolve any yellow salt that may have separated. Add ten cc. of magnesia mixture and stir well until the white crystalline precipitate of phosphate of magnesia and ammonia appears; about six cc. of ammonia (0.96 sp. gr.) are then added. Allow to stand two hours, filter upon a No. 2 ashless filter and wash with diluted ammonia (one part ammonia, 0.96 sp. gr., with three parts water).

About eighty cc. of this mixture are sufficient for washing the precipitate. It is advisable not to use more than this amount, as the same has a slightly solvent action upon the precipitate. The white precipitate must be rubbed loose from the sides of the beaker with rubber tubing on a glass rod.

The "magnesia mixture" is prepared by dissolving 110 grams of crystallized magnesium chloride together with 280 grams of ammonium chloride in 1300 cc. of water and adding 700 cc. of ammonia (0.96 sp. gr.) to the solution.

The filter with the well washed precipitate is ignited in a small weighed platinum crucible and weighed as magnesium pyrophosphate, care being taken that the ignited precipitate when weighed is white and uniform in color. Calculate the weight of phosphorus from this magnesium pyrophosphate.

If it be desired to estimate the phosphorus from the yellow precipitate (ammonio-molybdic phosphate) directly, proceed as follows: The yellow precipitate, when dried at 95° to 100° C., contains 1.63 per cent. of phosphorus. It must be washed with water containing one per cent., by volume, of nitric acid (1.2 sp. gr.) instead of the dilute molybdic solution. After drying it is transferred from the filter, by shaking and brushing, into a weighed watch-glass, or some other suitable vessel and weighed. When much phosphorus is present this method can be used with great accuracy, but when little the risk of loss is too great. Weighed filters must then be used.

The magnesia method is, however, undoubtedly the better of the two in general working.

When precipitating phosphoric acid with the molybdic acid solution it should be borne in mind that 100 cc. of the acid solution are required for the complete precipitation of one-tenth gram of phosphorus pentoxide containing 0.044 gram of phosphorus.

Many forms of agitation apparatus have been devised for the thorough precipitation of the ammonio-magnesium phosphate.

The apparatus of Spiegelbergs (Fig. 48), which is run by water power, is well adapted for the purpose of continued and violent agitation of the liquids.

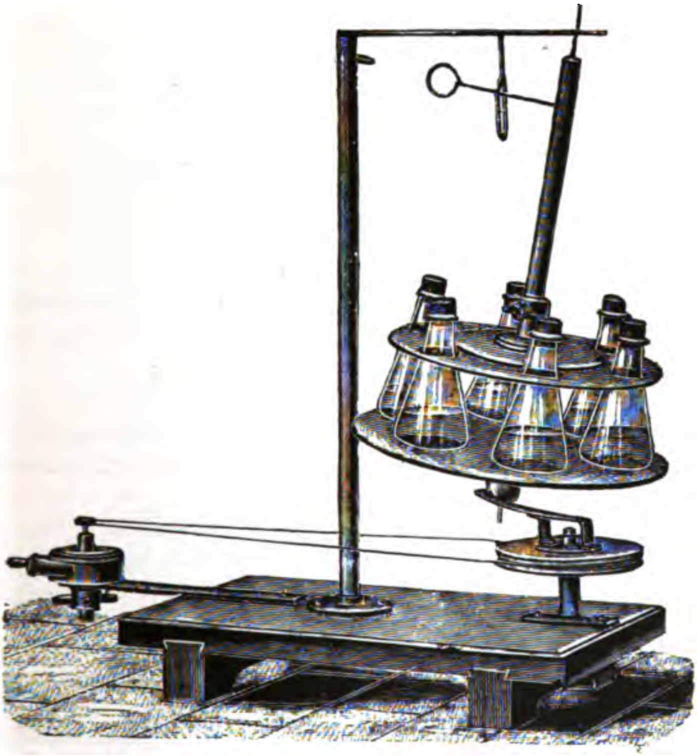

Fig. 48.

Volumetric Determination of Phosphorus in Iron and Steel.[1]

Put one gram of the steel in a ten or twelve ounce Erlenmeyer flask and add seventy-five cc. of nitric acid (1.13 sp. gr.). When solution is complete, boil one minute and then add ten

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[1] Method adopted by Motive Power Department of Penn. R. R. Co., Dudley and Pease, *J. Anal. Chem.*, 7, 108.

cc. of oxidizing potassium permanganate solution. Boil until the pink color disappears and manganese dioxide separates, remove from the heat and then add crystals of ferrous sulphate, free from phosphorus, with agitation until the solution clears up, adding as little excess as possible. Heat the clear solution to 185° F., and add seventy-five cc. of molybdate solution, which is at a temperature of 80° F., close the flask with a rubber stopper and shake five minutes, keeping the flask so inclosed during the operation that it will lose heat very slowly. Allow to stand five minutes for the precipitation to settle, and then filter through a nine cm. filter and wash with acid ammonium sulphate until the ammonium sulphide tested with the washings shows no change of color. Dissolve the yellow phospho-molybdate on the filter in five cc. of ammonia (sp. gr. 0.90), mixed with twenty-five cc. of water, allowing the solution to run back into the same flask and thus dissolve any yellow precipitate adhering to it. Wash until the washings and filtrate amount to 150 cc., then add ten cc. strong C. P. sulphuric acid and dilute to 200 cc. Now pass the liquid through a Jones reductor or its equivalent, wash and dilute to 400 cc., and then titrate in the reduction flask with potassium permanganate solution.

Apparatus and Reagents.—The apparatus required needs no especial comment, except perhaps the shaking apparatus and the modification of the Jones reductor. Accompanying cuts represent these two. The shaking apparatus is arranged to shake four flasks at a time, which is about all one operator can manipulate without the solutions becoming too cold. The cut is about one-twelfth the actual size of the apparatus. The flasks containing the solutions rest on a sheet of India rubber, about one-quarter inch thick and are held in position by the coiled springs as shown. There is a recess in the spring arrangement to receive the cork of the flask. Of course during use the door of the box is closed, the cut showing it open so that the interior may be seen. The modification reductor seems to work equally as well as the more elaborate apparatus. The cut is about one-fourth the actual size. As will be seen the tube is fitted with two rubber corks, the top one of which holds the funnel and the bottom one a small tube which also fits into the rubber cork in the flask. Next to the bottom cork in the tube is a disk of perforated platinum ; then about three fourths of an inch of clean white sand, then another perforated platinum disk and then the tube is nearly filled with powdered zinc. At least half the zinc may be used out before it is necessary to refill.

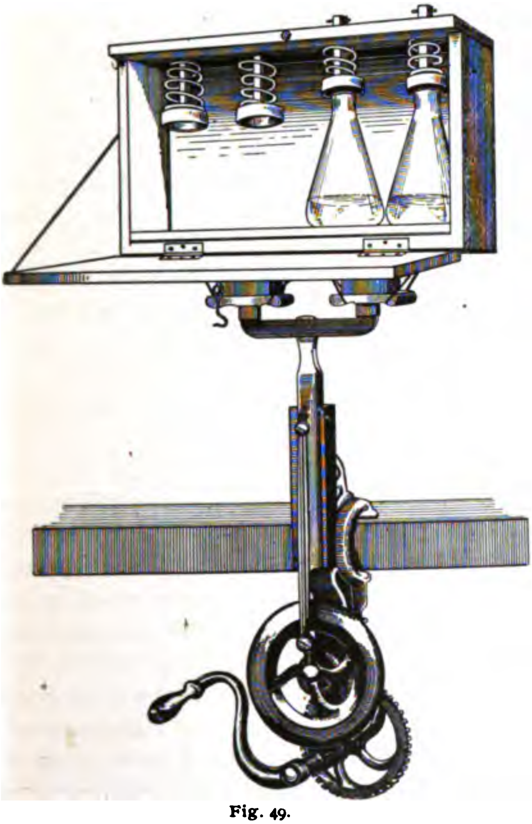

Fig. 49.

Fig. 50.

The oxidizing potassium permanganate solution is made as follows: To two liters of water add twenty-five grams of C. P. crystallized potassium permanganate and allow to settle before using. Keep in the dark.

The molybdate solution is made as follows: Dissolve 100 grams of molybdic acid in 400 cc. of ammonia (sp. gr. 0.96), and filter. Add the filtrate to one liter of nitric acid (sp. gr. 1.20). Allow to stand at least twenty-four hours before using.

The acid ammonium sulphate solution is made as follows: To one-half liter of water add 27.5 cc. of ammonia (sp. gr. 0.96) and then twenty-four cc. strong C. P. sulphuric acid and make solution up to one liter.

The potassium permanganate solution for titration is made as follows: To one liter of water add two grams of crystallized potassium permanganate and allow to stand in the dark not less than a week before using. Determine the value of this solution in terms of metallic iron. For this purpose 0.150 to 0.200 gram of iron wire or mild steel are dissolved in dilute sulphuric acid (ten cc. C. P. sulphuric acid to forty cc. water) in a long-necked flask. After solution is complete, boil five to ten minutes, then dilute to 150 cc., pass the liquid through a reductor and wash, make the volume up to 200 cc. Now titrate with the permanganate solution. Several determinations should be made. The figures showing the value of the permanganate solution in terms of metallic iron should agree to the hundredth of a milligram.

Calculations.—An example of all the calculations is given herewith. The soft steel employed in standardizing the potassium permanganate solution contains 99.27 per cent. metallic iron. 0.1498 gram of this contains (0.1498 × 0.9927) 0.1487064 gram Fe. This requires 42.99 cc. permanganate solution or one cc. = 0.003466 gram Fe. But the same amount of permanganate solution used up in producing the characteristic reaction in this amount of metallic iron, will be used up in reaction with 90.76 per cent. of the same amount of molybdic acid. Hence one cc. of the permanganate solution is equivalent to (0.003466 × 0.9076) 0.003145 gram of the molybdic acid. But in the yellow precipitate obtained as above described, the phosphorus is 1.90 per cent. of the molybdic acid. Hence one cc. of permanganate solution is equivalent to (0.003145 × 0.0190), 0.0000597 gram of phosphorus. If, therefore, in any sample of steel, tested as above, the yellow precipitate requires eight and six-tenths cc. of permanganate, the amount of phosphorus in that steel is (0.0000597 × 8.6) = 0.051 per cent.

Ten cc. of the "magnesia mixiure" are required for the same quantity of phosphorus pentoxide.

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XXI.

Classification of Steel.[1]

Classification of Steel Made by the Midvale Steel Company.

Class O. Carbon 0.1 to 0.2 per cent.
Approximate tensile strength from 55,000 to 65,000 pounds.
Class I. Carbon 0.2 to 0.3 per cent.
Approximate tensile strength from 65,000 to 75,000 pounds.
Class II. Carbon 0.3 to 0.4 per cent.
Approximate tensile strength from 75,000 to 85,000 pounds.
Class III. Carbon 0.4 to 0.5 per cent.
Approximate tensile strength from 85,000 to 95,000 pounds.
Class IV. Carbon 0.5 to 0.6 per cent.
Approximate tensile strength from 95,000 to 105,000 pounds.
Class V. Carbon 0.6 to 0.7 per cent.
Approximate tensile strength from 105,000 to 120,000 pounds.
Class VI. Carbon 0.7 to 0.8 per cent.
Approximate tensile strength from 120,000 to 135,000 pounds.
Class VII. Carbon 0.8 to 0.9 per cent.

On heats of this carbon and above, tensile strength is not considered, as they are generally used for spring steel and tool steel, in which the fitness of the material for the purpose wanted cannot be decided by the tensile strength of a test bar.

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[1] Prof. Coleman Sellers : *Stevens Indicator*, 11, *1894*, 88.

Class VIII.	Carbon	0.9 to 1.0 per cent.
Class IX	Carbon	1.00 to 1.10 per cent.
Class X	Carbon	1.10 to 1.20 per cent.

It is of course understood that while this classification holds good in a general way, the other chemical ingredients besides carbon, as well as treatment, may so effect the tensile strength that, while the percentage of carbon would place it in one class, other chemical ingredients or physical treatment may bring it (as far as tensile strength goes) into one of the other classes. As a general thing, it has been found that a high percentage of manganese, say above seven-tenths per cent., up to and including one per cent., will exert a much greater hardening influence on steels of high carbon than of steel below five-tenths per cent. in carbon; while the other chemical ingredients seem to exert a uniform hardening influence on all grades of steel.

The purposes for which the different classes of steel are recommended by the Midvale Steel Company, taking into consideration the many different specifications for the same purposes that are received, are as follows:

Classes I and II are used for propeller shafting, axles, and general machinery work. Also used for rifle-barrel steel, steel castings where toughness is the principal requirement, and finally, in the higher grades, where it approaches Class III, for gun tubes.

Class III is principally used for Pennsylvania Railroad axle and crank pins, and for parts of machinery where a high elastic limit is required. This class is recommended for axles and crank pins, and, where the choice is left with the makers, they invariably use it for this purpose. It is a class which, in their opinion, is best suited for steel forgings of all descriptions, with the conditions, however, that the forgings should be thoroughly annealed. If this is not done, the lower class is preferable, as the strains left in the forging are not apt to be injurious in the lower carbon steel. This class is also used for gun forgings, jackets and hoops, the high requirements as to elastic limit making it necessary to have a good percentage of carbon.

Class IV is used also principally for gun forgings and for large locomotive tires.

Class V is used principally for tires for freight service and car wheels, and for forgings for air vessels for torpedoes, and also for steel castings where greater wear is desirable, such as hammer dies, roll pinions, etc.

Class VI is used mostly for surgical instruments and grinding machinery.

Class VII is used for spring steel.

Classes VIII, IX and X are used for various grades of spring and tool steel, the highest grades being used for cutting tools and the lower grades for chisels, reamers, etc.

It is necessary to remember in this classification, that while the carbon and tensile strength governs the classes, the chemical composition of the different heats that come under one class varies considerably. In the case of ordinary machinery steel and tires, the makers endeavor to keep the phosphorus limit below 0.06 per-cent. This is the case also with their steel castings. In gun forgings, on the other hand, their phosphorus limit is below 0.03 per cent., as well as in tool steel and spring steel.

At the present moment, the greatest interest is taken in the magnetic qualities of steel, as compared with the best Norway iron; and from recent experiments it will be seen that conclusions can not be drawn with safety from a few experiments, particularly in regard to the alloys of various metals with steel. The statement has been broadly made that a large percentage of nickel introduced into steel castings destroyed the magnetic qualities of the steel to such an extent as to make this alloy particularly desirable or useful for the bolts that clamp the punchings of the armature in the dynamo together, the general idea being that these foreign substances were all acting injuriously. Some recent experiments have been made by the Bethlehem Iron Company, bearing upon the dynamos that are to be made for Niagara, which seem to show that when a small quantity of nickel only is used, the magnetic qualities are improved to such a degree as to make its employment advisable, making nickel steel, properly prepared, higher in its capability of magnetization than even the best Norway iron. This statement does not hold good in all degrees of excitation, but is said to be particularly good at the amount of excitation to which field magnets are usually subjected.

Mr. L. B. Stillwell, in his examination of this metal, concludes a report on the subject with the words: "I am emphatically of the opinion that no better material can be secured." The effect of the mixture of foreign substances with steel is one that is worthy of the most careful attention of the students of technical colleges, and would form an admirable subject for a thesis, as the experiments to be reliable need not involve great cost, and would give opportunity for a considerable display of ingenuity in devising methods of making the tests, and the manner of showing the results by graphical methods.

Steel plate for locomotive use requires the carbon to be not under 0.15 per cent. nor over 0.20 per cent;[1] phosphorus 0.03 per cent. to 0.04 per cent.; manganese 0.35 per cent. to 0.50 per cent.; silicon 0.025 per cent. to 0.04 per cent.; sulphur 0.02 per cent. to 0.04 per cent.; copper (if any) not over 0.04 per cent.

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[1] *The Engineer*, March 30, 1895.

Classification of Iron and Steel.—Kent.

Generic term	IRON							
How obtained.	CAST, or obtained from a fluid mass						WROUGHT, or welded from a pasty mass.	
Distinguishing quality	Non-malleable.	Malleable					Will harden	Will not harden
Species	CAST IRON.		CAST STEEL.				WROUGHT IRON.	WROUGHT STEEL[2]
Varieties	Ordinary castings.	Mall'ble cast iron (made from No. 1, by annealing in oxides.)	Crucible steel.	Bessemer steel.	Open hearth steel.	*Mitis.*	Obtained by direct process from ores. Catalan, Siemens, Chenot and other process irons. Obtained by indirect process from cast iron. Finery, Lancashire and puddled irons. Com'on wrought iron.	Obtained by direct or indirect process. German Shear Blister and Puddled Steel.[2]
No.	1	2	3	4	5	6[1]	7	8[2]

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[1] No. 6. *Mitis* is the name given a new product (having the same general qualities and produced by the same processes as soft cast steels, and therefore classified with them), distinguised by great fidelity in pouring and casting, which is secured by adding an alloy of aluminum to the metal before pouring.

[2] No. 8. Wrought steel including German, puddled, blister and shear steel is nearly an obsolete product, having been replaced in commerce by those qualities of cast steel which will harden and temper.

XXII.

Determination of Aluminum in Iron and Steel.

The direct determination of aluminum in iron and steel is somewhat difficult, especially if the amount of aluminum be small.

Drown[1] describes a process which gives good results, as follows :

Dissolve five to ten grams of iron or steel in sulphuric acid, evaporate until white fumes of sulphuric anhydride begin to come off, add water, heat until all the iron is in solution, filter off the silica and carbon, and wash with water acidulated with sulphuric acid. Make the filtrate nearly neutral with ammonia, and add to the beaker in which the electrolysis is to be made, about 100 times as much mercury as the weight of iron or steel taken. The bulk of the solution should be from 300 to 500 cc. Connect with the battery or dynamo current in such a way that about two amperes may pass through the solution over night. This is generally accomplished by using three lamps (thirty-two candle power) arranged in parallel on an Edison circuit. In the morning the solution is tested for iron, and, if necessary, the electrolysis is continued after adding enough ammonia to neutralize the acid that has been set free by the deposition of the iron. The progress of the operation may be observed by the changing color of the solution. At first it becomes darker in color near the anode; after five or six hours it is nearly colorless, and finally becomes pink, from the formation of permanganate.

When the solution gives no test for iron, it is removed from the beaker with a pipette while the current is still passing. When as much has been removed as possible without breaking the current, water is added, and the operation continued until the acid has been so far diluted that there is no danger of dissolving iron from the mercury. The anode is now taken out and the mercury washed with water until the last traces of the solution have been removed from it.

After filtering, to remove any flakes of manganese dioxide

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[1] *J. Anal. Appl. Chem.*, 5, 631.

which may be suspended in the solution, sodium phosphate is added in excess and ten grams of sodium acetate. The solution is now made nearly neutral with ammonia and boiled for not less than forty minutes. The precipitate of aluminum phosphate is then filtered off, ignited, and weighed. It should be white after ignition. If it has more than the faintest shade of color it must be dissolved by fusing with acid potassium sulphate, in a platinum crucible, and again electrolyzed for two or three hours. The second precipitate has been found to be always white without a trace of iron. The precipitate of aluminum phosphate, produced as above, does not always have the composition $Al_2O_3.P_2O_5$. It is more nearly expressed by the formula $7Al_2O_3.6P_2O_5$, containing 24.14 per cent.

The following table gives the results obtained in determining by the above process the aluminum added in known amounts to solutions of steel:

Steel taken. Grams.	Per cent. of aluminum added.	Per cent. of aluminum found.
5	0.39	0.36
5	0.39	0.38
5	0.39	0.38
5	0.39	0.38
5	0.39	0.37
5	0.043	0.045
5	0.043	0.041
5	0.043	0.049
5	0 043	0.048
10	0.027	0.015
10	0.200	0.160
10	0.046	0.044
5	0.085	0.088

A blank experiment with the same steel, without the addition of any aluminum, gave a precipitate of aluminum phosphate equivalent to 0.004 per cent. of aluminum. It might be thought that the process would be simplified by reducing the iron to the state of protoxide, and then precipitating alumina as basic acetate, subsequently removing by electrolysis the small amount of iron precipitated with the alumina. A number of experiments proved, however, that this modification not only gave less accurate results, but involved much more work than the precipitation of all of the iron by electrolysis.

Method of Carnot.

Treat ten grams of the iron or steel in a platinum dish covered with platinum foil, with hydrochloric acid, and when solution is complete, dilute and filter into a flask, washing the carbon, silica, etc., on the filter, thoroughly with distilled water. Neutralize the solution with ammonia and sodium carbonate, but see that no permanent precipitate is formed; then add a little sodium hyposulphite, and when the liquid, at first violet, becomes colorless, two or three cc. of a saturated solution of sodium phosphate and five or six grams of sodium acetate dissolved in a little water. Boil the solution for about three-quarters of an hour, or until it no longer smells of sulphurous acid. Filter and wash the precipitate of aluminum phosphate mixed with a little silica and ferric phosphate, with boiling water. Treat the precipitate on the filter with hot dilute hydrochloric acid, allow the solution to run into a platinum dish, evaporate to dryness, and heat at 100° C. for an hour to render the silica insoluble. Dissolve in hot dilute hydrochloric acid, filter from the silica, dilute to about 100 cc. with cold water, neutralize as before, add a little hyposulphite in the cold, then a mixture of two grams of sodium phosphate and two grams of sodium acetate, boil until all smell of sulphurous acid has disappeared, filter, wash, ignite, and weigh as $Al_2O_3.P_2O_5$, which contains 22.18 per cent. of aluminum.[1]

References: "A Rapid Method for the Determination of Aluminum in Iron and Steel." *Chem. News*, 61, 313.

"On the Determination of Minute Quantities of Aluminum in Iron and Steel." By John E. Stead, F. I. C., *J. Soc. Chem. Industry, 1889*, p. 956.

XXIII.

Determination of Sulphuric Acid and Free Sulphur Trioxide in Fuming Nordhausen Oil of Vitriol.

As this acid fumes immediately upon exposure to the air, also rapidly absorbing moisture, great expedition must be exercised in obtaining the samples for analysis.

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[1] *J. Anal. Chem.*, 5, 178.

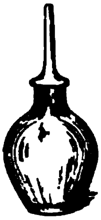

Fig. 51.

Select a small picnometer (Fig. 51), weight about eight grams, and determine its weight with great accuracy. Insert a pipette into the Nordhausen acid, and without suction allow about two grams of the acid to run into the pipette. Remove the stopper of the picnometer, insert the lower end of the pipette into it, allow the acid to flow, remove the pipette and insert the stopper of the picnometer. Weigh the picnometer and acid carefully to the fourth decimal; then drop it into a tall beaker (capacity 800 cc.) containing about 500 cc. of distilled water and cover with a watch glass; remove the stopper of the picnometer at the moment the latter is dropped into the water. Too much acid should not be used, three grams being the maximum amount.

Determine the amount of acid present by titration with a solution of soda, which will give the total sulphur trioxide, but as Nordhausen acid is composed of varying amounts of a mixture of sulphuric acid and sulphur trioxide, it will be well to explain the method in detail.

Picnometer and Nordhausen acid	= 8.7210	grams.
Picnometer	= 7.6320	"
Nordhausen acid	= 1.0890	"

Amount of soda solution required to neutralize 1.089 grams of the acid = 28.7 cc.

One cc. of the soda solution is equivalent to 0.0401 gram sulphuric acid or 0.0327 gram sulphur trioxide.

The acid therefore contains 86.2 per cent. of sulphur trioxide and 13.8 per cent. of water.

To determine the proportions of sulphur trioxide and sulphuric acid the following formulas are used:

Let $x = H_2SO_4$ in the acid.

$y = SO_3$ " " "

$$x + y = 100.$$

$$\frac{80}{98} x + y = 86.2$$

$$98x + 98y = 9800.$$

$$80x + 98y = 8447.6$$

$$18x \qquad = 1352.4$$

$x = 75.1$ per cent. of H_2SO_4 in the acid.
$100 - 75.1 = 24.9$ per cent. of SO_3 in the acid.
$y = 24.9$ per cent of SO_3 in the acid.

$$\underset{(x)}{75.1} + \underset{(y)}{24.9} = 100.$$

Nordhausen acid often contains small amounts of sulphur dioxide. This should be boiled out of the water before titration with the soda solution.

XXIV.

Determination of Manganese in Iron and Steel.

Manganese can be determined accurately in iron and steel colorimetrically, gravimetrically or volumetrically. The latter method is in general use as being expeditious.

For the gravimetric and volumetric methods the initial treatment may be the same, that is, solution of the steel in nitric acid; the precipitation of the oxide of manganese by means of the nitric acid and potassium chlorate, and its filtration and separation.

Five grams of the steel are transferred to a No. 5 beaker and 150 cc. of nitric acid (sp. gr. 1.2) added. After solution of the iron and concentration to about 100 cc., there is added fifty cc. nitric acid (sp. gr. 1.42) and the boiling continued till the bulk of the liquid amounts to about 100 cc. To this is added crystals of potassium chlorate (not over three grams) *gradually*, and the boiling continued until no more fumes of chlorous gas are emitted. Allow to cool, add twenty-five cc. nitric acid (sp. gr. 1.42) and filter upon an asbestos filter, washing twice with strong nitric acid and five times with cold water. Transfer the filter and contents to a beaker and treat *a*, for gravimetric determination, or *b* for volumetric determination of the manganese.

a. Add seventy-five cc. hydrochloric acid (strong) and boil; the manganese dioxide is dissolved. The solution is diluted with water and the asbestos separated therefrom by filtration upon a No. 4 filter, and well washed. The filtrate is made faintly alkaline with ammonia, then to acid reaction with acetic acid, and boiled. Filter off any basic acetate of iron that may be present, and to the filtrate add ammonium hydroxide to alkaline reaction and then bromine (not over one cc.); shake well, set aside two hours, then boil, filter, dry, ignite, and weigh as Mn_3O_4. Consult scheme XIII.

b. Instead of dissolving the manganese dioxide in hydrochloric acid, as in *a*, it is dissolved in a measured amount of standard acid solution of ferrous sulphate, and the excess of ferrous sulphate determined by a standard solution of potassium bichromate. The ferrous sulphate solution is made by dissolving twenty grams crystallized ferrous sulphate in 1600 cc. water and adding thereto 400 cc. of sulphuric acid (sp. gr. 1.5).

The bichromate solution is made by dissolving ten grams of potassium dichromate in 1000 cc. water. One cc. of the ferrous sulphate solution corresponds to 0.011 gram of iron, that is, it will oxidize the amount of ferrous sulphate to ferric sulphate that corresponds to 0.011 gram of iron. One cc. of the bichromate solution corresponds to 0.0054 gram manganese.

The manganese dioxide precipitate, obtained from the five grams of steel, is dissolved in 100 cc. of the acid ferrous sulphate solution; it is then titrated with bichromate solution until a drop of the liquid placed on a porcelain slab and brought in contact with a drop of *fresh* dilute solution of potassium ferricyanide shows no blue or green color, but a faint brown color, (Scheme VIII) indicating complete oxidation.

The amount of bichromate that would be required to oxidize the total iron in the 100 cc. would be 18.1 cc., but in this experiment 15.1 cc. were required, showing that the oxidizing action of three cc. of the bichromate solution had been supplanted by the action of the manganese dioxide. Since three cc. of the bichromate corresponds to 0.0162 gram manganese dioxide, and this amount is obtained from five grams of the steel, the per cent. of manganese dioxide will be $\frac{0.0162 \times 100}{5} =$ 0.324 per cent. Some chemists prefer the use of a solution of potassium permanganate instead of potassium bichromate. (Consult, *Trans. American Inst. Mining Engineers*, 10, 100.)

The color method may be stated briefly as follows: In a test-tube, similar to that used for the estimation of carbon, place two-tenths gram of the sample to be tested, and in a like tube the same quantity of a standard steel, in which the manganese has been carefully determined by weight. To each add five cc. nitric acid (sp. gr. 1.20), and boil in a beaker of hot water until solution is complete. Cool the tubes, and to each add an equal bulk, about two cc. of water; replace in the beaker, and, after boiling for a few minutes, add an excess of lead peroxide, which must be free from manganese, and ten drops of nitric acid (sp. gr. 1.42.) After boiling for four minutes the tubes are withdrawn and placed in a beaker of cold water. When the peroxide of lead has completely settled, transfer two cc. of the clear supernatant liquid of the standard solution to the graduated tube used in the colorimetric estimation of carbon, dilute to five cc. with cold water, mix. In a similar tube place the same quantity of the solution of the sample which is being tested, diluting with water until its color is of the same intensity as that of the standard. Read off the number of cc. to which dilution is carried, from which, by a simple calculation, the percentage is easily determined.[1]

Textor's Method for the Rapid Determination of Manganese in Steel.

To one-tenth gram of steel, in a No. 2 beaker, add fifteen cc. of nitric acid (sp. gr. 1.20); boil until the brown oxides of nitrogen are gone; add fifteen cc. of hot water, and while boiling introduce one-half gram of lead peroxide. Boil three minutes after the addition of the lead peroxide, filter through asbestos, and wash with water containing two per cent. nitric acid (sp. gr. 1.20). Titrate with a solution of arsenious acid till the pink color is gone; each cubic centimeter of solution equals one-tenth per cent. of manganese.

Precautions.—The brown fumes must all be expelled before adding water, otherwise low results may be expected. Before filtering, the asbestos must be treated with nitric acid. For steels containing 0.75 per cent. of manganese, one-half gram or more lead peroxide should be added, and the solution, after the

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[1] J. J. Morgan: *Chem. News*, 56, 82.

addition of the lead, should be boiled not less than three minutes, otherwise low results may be obtained. To secure rapid filtration, a special filter is required. It may be constructed as follows: Fill a two and one-half inch funnel one-third to a half full with pieces of glass rod one-quarter to one-half inch long; on this place a disk of platinum foil fitting the funnel at the point where the disk rests on the broken glass. The platinum disk is perforated by means of a pin, over its whole surface; the rough side is turned down. Pour suspended asbestos upon the foil till a layer is formed one-half inch in thickness. When the filter becomes clogged and works slowly, the thin layer of lead peroxide can be removed by carefully scraping with a wire, a fresh surface of asbestos thereby becoming exposed.

For the arsenic solution, twenty grams of arsenic trioxide in powder and sixty grams of sodium carbonate are dissolved in 750 cc. of hot water, filtered and diluted to 2000 cc. An equivalent amount of sodium arsenite may be conveniently taken. Of this solution, 87.5 cc. are diluted to 2500 cc. and tested with a steel containing a known percentage of manganese.[1]

References: "Colorimetric Estimation of Manganese in Steel." By B. W. Cheever, *J. Anal. Chem.*, 1, 88.

"Volumetric Determination of Manganese." By J. Pattison, *J. Chem. Soc.*, 35, 365.

"Method for the Rapid Determination of Manganese in Slags, Ores, Etc." By F. G. Myhlertz, *J. Anal. Chem.*. 4, 267.

XXV.

Technical Determination of Zinc in Ores.

Prepare a solution of potassium ferrocyanide by dissolving forty-four grams of the pure salt in distilled water and diluting to one liter. Standardize as follows:[2] Dissolve 200 milligrams of pure zinc oxide in ten cc. of strong pure hydrochloric acid. Add seven grams of C. P. ammonium chloride, and about 100 cc. of boiling hot water. Titrate the clear liquid with the ferrocyanide solution until a drop, tested on a porcelain plate with a drop of a strong aqueous solution of uranium acetate, shows a brown tinge. About sixteen cc. of

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[1] Engineers' Society of Western Pa., Trans., 1892.

[2] Method of von Schulz and Low.

ferrocyanide will be required, and accordingly this amount may be run in rapidly before making a test, and then the titration finished carefully by testing after each additional drop of ferrocyanide. As soon as a brown tinge is obtained note the reading of the burette, and then wait a minute or two and observe if one or more of the previous tests do not also develop a brown tinge. Usually the end-point will be found to have been passed by a test or two, and the proper correction must then be applied to the burette reading. Finally make a further deduction from the burette reading of the amount of ferrocyanide required to produce a brown tinge under the same conditions when no zinc is present. This correction is about two drops, or 0.14 cc. Two hundred milligrams of zinc oxide contain 160.4 milligrams of zinc, and one cc. of the above standardized solution will equal about 0.01 gram of zinc, or about one per cent., when one gram of ore is taken for assay.

Prepare the following solutions for the assay of ores:

A saturated solution of potassium chlorate in nitric acid, made by shaking an excess of crystals with the strong acid in a flask. Keep the solution in an open flask.

A dilute solution of ammonium chloride containing about ten grams to the liter; for use heat to boiling in a wash bottle.

Take exactly one gram of the ore and treat in a three and one-half inch casserole with twenty-five cc. of the above chlorate solution. Do not cover the casserole at first, but warm gently until any violent action is over and greenish vapors have ceased to come off. Then cover with a watch-glass and boil to complete dryness, but avoid overheating and baking. Cool sufficiently and add seven grams of ammonium chloride, fifteen cc. strong ammonia water, and twenty-five cc. hot water. Boil the covered mixture one minute and then, with a rubber-tipped glass rod, see that all solid matter on the cover, sides and bottom of casserole is either dissolved or disintegrated. Filter into a beaker and wash several times with the hot ammonium chloride solution. A blue colored solution indicates the presence of copper. In that case add twenty-five cc. strong pure hydrochloric acid and about forty grams of granulated test-lead. Stir the lead about in the beaker until the liquid has become perfectly colorless and then a little longer to make sure that all the copper is precipitated. The solution, which should be quite hot, is now ready for titration. In the absence of copper the lead is omitted and only the acid added. About one-third of the solution is now set aside, and the main portion is titrated rapidly with the ferrocyanide until the end-point is passed, using the uranium indicator as in the standardization. The greater part of the reserved portion is now added, and the titration continued with more caution until the end-point is again passed. Then add the remainder of the reserved portion and finish the titration carefully, ordinarily by additions of two drops of ferrocyanide at a time. Make corrections of this final reading of the burette as in the standardization.

Gold, silver, lead, copper, iron, manganese, and the ordinary constituents of ores do not interfere with the above scheme. Cadmium behaves like zinc. When known to be present it may be removed, together with the copper, by the proper treatment with hydrogen sulphide, and the titration for zinc may be made upon the properly acidified filtrate without the removal of the excess of gas.

XXVI.

Sodium Cyanide as a Component of Potassium Cyanide.

The valuation of potassium cyanide for commercial purposes, is dependent upon the amount of cyanogen present, the salt being rated from "thirty per cent. cyanide" to "ninety-eight per cent. cyanide"—the former selling for twenty cents and the latter for sixty cents per pound. The determination of the percentage of cyanogen is usually made by titration with semi-normal silver solution, and in chemical manufactories where potassium cyanide is made, generally constitutes the entire analysis. Potassium cyanide, when pure, contains forty per cent. of cyanogen; "ninety-eight per cent." would, therefore, indicate 39.2 per cent. of cyanogen, and "thirty per cent.," twelve per cent. of cyanogen. An analysis of a sample of the former gave by titration 42.33 per cent. of cyanogen, or a rating of 105.87 per cent. of potassium cyanide. This result immediately showed that another base than potassium was present, and one also whose combining weight was less. Sodium being indicated by qualitative analysis, a quantitative analysis of the sample was necessary to determine the proportions of potassium and sodium combined with the cyanogen.

The method adopted was as follows: The cyanide was weighed, transferred to a platinum capsule, sufficient water added for solution, then dilute sulphuric acid in excess and contents evaporated to dryness and ignition to constant weight. This represented sulphates of potassium and sodium, and after solution in water and acidifying with hydrochloric acid, the sulphuric acid was precipitated and weighed as barium sulphate and calculated to SO_3.

These determinations gave a method of obtaining the proportions of potassium and sodium in the weighed alkaline sulphate as follows:

94.2 parts K_2O require 80 parts SO_3 for K_2SO_4
62.0 " Na_2O " 80 " SO_3 " Na_2SO_4

Let G = weight of sulphates.
" x = " " K_2O.
" y = " " Na_2O

$$\text{Then } G = x + y + \frac{80}{94.2}x + \frac{80}{62}y$$

$$\text{Or, } G = x + y + 0.85\,x + 1.29\,y$$

$$x + y = G - SO_3$$

$$y = \frac{1.85\,SO_3 - 0.85\,G}{0.4387}$$

$$x = G - (SO_3 + y)$$

Having obtained the values of potassium oxide and sodium oxide, they are calculated to potassium and sodium. These weights are multiplied by 100 and divided by the weight of cyanide taken, the results being the percentages of potassium and sodium respectively in the cyanide. If to these results is added the percentage of cyanogen, as determined by titration with semi-normal silver solution, the analysis is completed.

A sample of the cyanide above mentioned as containing sodium as well as potassium, gave the following :

Amount of salt taken for analysis, 1.519 grams.

Platinum capsule and alkaline sulphates	46.625	grams.
" "	44.573	"
$K_2SO_4 + Na_2SO_4$	2.052	"
Crucible and $BaSO_4$	25.165	"
"	22.306	"
$BaSO_4$	2.859	"

Equivalent to 0.981 gram SO_3. Cyanogen by titration was 42.33 per cent.

$$Na_2O = \frac{1.85\,(0.981) - 0.85\,(2.052)}{0.4387} = 0.161 \text{ gram.}$$

$$= 0.120 \text{ gram sodium, or } 7.90 \text{ per cent.}$$

$$K_2O = 2.052 - (0.981 + 0.161) = 0.910 \text{ gram.}$$

$$= 0.755 \text{ gram potassium, or } 49.70 \text{ per cent.}$$

Resulting:

Sodium	7.90	per	cent.
Potassium	49.70	"	"
Cyanogen	42.33	"	"
Undetermined	0.07	"	"
Total	100.00	"	"

Equivalent to:

Sodium cyanide	16.90	per	cent.
Potassium cyanide	82.83	"	"
Difference	0.20	"	"
Undetermined	0.07	"	"
Total	100.00	"	"

This cyanide of potassium and sodium (though marked "potassium cyanide, ninety-eight per cent.") is sold at a lower rate than the "ninety-eight per cent. potassium cyanide," and for many purposes is superior, as it contains a higher percentage of cyanogen. An examination of the formula for its manufacture shows that it can be made at a less cost than the potassium cyanide alone. Potassium ferrocyanide, or sodium ferrocyanide when heated in covered crucibles is converted into potassium or sodium cyanide, iron carbide and nitrogen:

$$2K_4Fe(CN)_6 = 8KCN + 2FeC_2 + N_4$$

$$2Na_4Fe\,(CN)_6 = 8NaCN + 2FeC_2 + N_4$$

100 pounds of potassium ferrocyanide, at thirty cents per pound, produces 70.63 pounds of potassium cyanide, ninety-eight per cent., at a cost of forty-two cents per pound; and 100 pounds of sodium ferrocyanide, at twenty cents per pound, produces 64.47 pounds of sodium cyanide, ninety-eight per cent., at a cost of thirty-one cents per pound.

If a mixture composed of 117 pounds of potassium ferrocyanide and twenty-six pounds of sodium ferrocyanide be heated in covered crucibles, the resulting compound, weighing 100 pounds, will closely approximate, in composition, the sample submitted.

XXVII.

The Chemical and Physical Examination of Portland Cement.

The enlarged consumption of Portland cement in this country during the past few years has caused the subject of its chemical and physical properties to receive increased consideration. Not only has the consumer been directly interested, that the cements used should stand special tests, but the attention of the manufacturer has been drawn in the same direction, resulting in improvements in methods of production.

A number of causes have prevented the use of American Portland cements in the home market, one of the chief being that the imported German cements always give higher physical tests when made by the German methods of testing than the American cements under the American system of testing. There are a number of American Portland cements fully as good as the best German cements, and have shown fully as high tensile strength when tested by the same methods.

These differences in results are not due entirely to the cements, but rather to the methods in use in the different countries for testing them, for Portland cements cannot vary much in their chemical composition without losing their value.

The limit of variation is as follows:

CaO	58.0 to 67.0	per cent.[1]
SiO_2	20.0 to 26.0	“ “
Al_2O_3	5.0 to 10.0	“ “
Fe_2O_3	2.0 to 6.0	“ “
MgO	0.5 to 3.0	“ “
SO_3	0.5 to 2.0	“ “

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[1] E. Candlot: *Étude practique sur le Ciment de Portland*, (Paris, 1886).

After manufacture it is practically Ca_3SiO_5, and is quite distinct from another product made and largely consumed here called "hydraulic cement."

Experience has shown that Portland cements containing over two per cent. of magnesia (MgO) are inferior in lasting qualities, and by the gradual absorption of water produce cracking and disintegration.[1]

Calcium carbonate ($CaCO_3$), formed by the absorption of carbon dioxide by the lime in the cement after manufacture, is another injurious compound found in cements containing more lime than sufficient to unite with the silica to form tri-silicate of lime. This carbonate of lime gradually produces seams and fractures after the setting of the cement. The "Ecole Nationale," of Paris, rejects all cements containing over one and five-tents per cent. of sulphuric acid. Thus, if upon chemical analysis, magnesia is found present in amounts over two per cent. carbonic and sulphuric acids in amounts over one and one-half per cent., *the cement can be condemned at once without any mechanical tests.* Therefore, it is evident that a careful test of a Portland cement requires: (1) a chemical analysis to determine the proportion of the ingredients, and (2) the mechanical or physical tests to determine fineness, tensile strength, and resistance to crushing.

The following scheme is arranged to show the method of making a cement analysis:

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[1] *Compt. rend.*, May, 1886.

Analysis of Portland Cement.

Weigh out 2 grams of the finely powdered and dried material, transfer to a 6-inch porcelain capsule, add 50 cc. HCl, 5 cc. HNO_3, and evaporate to dryness; add 25 cc. HCl, 100 cc. H_2O, boil and filter into a ½ liter flask; wash well; make solution up to containing mark and thoroughly mix.

<table>
<tr>
<td colspan="2" rowspan="2">(1) Residue.—Dry, ignite, fuse in platinum crucible with Na_2CO_3; cool, dissolve in water, acidify with HCl, evaporate to dryness in 4-inch porcelain capsule; take up with HCl and water: boil, filter and wash.</td>
<td colspan="7">(2) Solution.[1]—Take 1 sample of 100 cc., transfer to a 300 cc. beaker, make alkaline with NH_4OH, warm, filter, and wash.</td>
</tr>
<tr>
<td colspan="2">(3) Residue, Fe_2O_3, Al_2O_3. Dry, ignite and weigh as such. Then fuse in silver dish with KOH. Treat with water, boil, and filter.</td>
<td colspan="4">(4) Solution.—Add $(NH_4)_2C_2O_4$ in slight excess, set aside 4 hours, filter, wash with water containing ½ its bulk of NH_4OH.</td>
<td rowspan="4">SO_3.—50 cc. of the solution are transferred to a 100 cc. beaker; solution of $BaCl_2$ is added in slight excess; boil and set aside for 6 hours; filter, wash, dry, ignite and weigh as $BaSO_4$, and calculate to SO_3.</td>
</tr>
<tr>
<td rowspan="3">Residue, SiO_2. Dry, ignite and weigh as SiO_2.</td>
<td rowspan="3">Solution.—Make alkaline with NH_4OH: warm, filter, wash. (Neglect filtrate.) Residue is Al_2O_3. Dry, ignite, and weigh as Al_2O_3.</td>
<td rowspan="3">Residue, Fe_2O_3. Dry, ignite, and weigh as Fe_2O_3. Subtract this weight from weight in (3). The difference is Al_2O_3.</td>
<td rowspan="3">Solution, contains Al_2O_3. If it is desired to weigh this. Acidify this solution with HCl, then make faintly alkaline with NH_4OH: boil, filter, wash well. The precipitate is dried, ignited and weighed as Al_2O_3.</td>
<td rowspan="3">(5) Residue is CaC_2O_4. Dry, ignite over blast lamp to constant weight. Weigh as CaO.</td>
<td colspan="3">(6) Solution.—Evaporate to dryness in a platinum capsule, ignite to expel all ammonia salts; cool, add 50 cc. H_2O, boil, filter and wash.</td>
</tr>
<tr>
<td rowspan="2">(7) Residue is MgO. Dry, ignite and weigh as MgO.</td>
<td colspan="2">(8) Solution.—Transfer to a weighed platinum capsule, add a few drops of H_2SO_4 and evaporate to dryness; ignite to constant weight. This weight represents Na_2SO_4, K_2SO_4, $MgSO_4$. Dissolve in 50 cc. of H_2O, mix well and divide into two portions of 25 cc. each.</td>
</tr>
<tr>
<td>1st Portion.—Add a few drops of HCl, then make alkaline with NH_4OH, and precipitate the Mg with solution of Na_2HPO_4. Set aside 3 hours, filter, wash with ammoniated water, dry, ignite and weigh as $Mg_2P_2O_7$, and calculate to $(MgSO_4)_2$. This weight multiplied by 2, is subtracted from weight of total sulphates in (8). Calculate $MgSO_4$ to MgO.</td>
<td>2d Portion.—Add solution of $PtCl_4$ in slight excess, a few drops of HCl and evaporate to dryness on the water-bath. Determine weight of K_2PtCl_6 on tared filters and calculate to K_2SO_4. Multiply this by 2 and subtract weight from total sulphates. The weights of K_2SO_4 and $MgSO_4$ subtracted from total sulphates gives the remainder as Na_2SO_4. Calculate K_2SO_4 to K_2O. Calculate Na_2SO_4 to Na_2O.</td>
</tr>
<tr>
<td>SiO_2.</td>
<td>Al_2O_3</td>
<td>Fe_2O_3.</td>
<td>Al_2O_3.</td>
<td>CaO.</td>
<td>MgO.</td>
<td>K_2O.</td>
<td>Na_2O.</td>
<td>SO_3.</td>
</tr>
</table>

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[1] Manganese oxide is seldom found in Portland cement. If qualitative analysis has indicated its presence, the iron oxide and alumina must be separated out by sodium acetate process in (2) of above scheme. The filtrate containing the manganese is treated with bromine, the MnO_2 filtered off, and the filtrate treated as directed in (4).

$$\frac{\text{Weight of SiO}_2 \times 100}{2} = \text{per cent. SiO}_2.$$

(1) Crucible + SiO_2 .... 11.205 grams.
Crucible .... 10.721 “
SiO_2 = 0.484 “

$$\frac{0.484 \times 100}{2} = 24.20 \text{ per cent. SiO}_2.$$

$$\frac{\text{Weight Al}_2\text{O}_3 \times 100}{2} = \text{per cent. Al}_2\text{O}_3 \text{ in the insoluble residue.}$$

(1) Crucible + Al_2O_3 .... 10.743 grams.
Crucible .... 10.721 “
Al_2O_3 = 0.022 “

$$\frac{0.022 \times 100}{2} = 1.10 \text{ per cent. Al}_2\text{O}_3.$$

$$\frac{\text{Weight of Fe}_2\text{O}_3 \times 2.5 \times 100}{2} = \text{per cent. Fe}_2\text{O}_3,$$

(2) Crucible + Fe_2O_3 .... 10.745 grams.
Crucible .... 10.721 “
Fe_2O_3 = 0.024 “

$$\frac{0.024 \times 2.5 \times 100}{2} = 3.00 \text{ per cent. Fe}_2\text{O}_3.$$

$$\frac{\text{Weight of Al}_2\text{O}_3 \times 2.5 \times 100}{2} = \text{per cent. Al}_2\text{O}_3.$$

(2) Crucible + Al_2O_3 .... 10.762 grams.
Crucible .... 10.721 “
Al_2O_3 = 0.041 “

$$\frac{0.041 \times 2.5 \times 100}{2} = 5.12 \text{ per cent. Al}_2\text{O}_3.$$

$$5.12 + 1.10 = 6.22 \text{ per cent. Al}_2\text{O}_3.$$

$$\frac{\text{Weight of CaO} \times 2.5 \times 100}{2} = \text{per cent. CaO.}$$

(5) Crucible + CaO .... 11.2223 grams.
Crucible .... 10.7210 “
CaO = 0.5013 “

$$\frac{0.5013 \times 2.50 \times 100}{2} = 62.67 \text{ per cent. CaO.}$$

(7) Crucible + MgO .... 10.725 grams.
Crucible .... 10.721 “
MgO = 0.004 “

(8)

$\left.\begin{array}{l}\text{Alkaline sulphates}\\ K_2SO_4.Na_2SO_4.MgSO_4\end{array}\right\}$ +platinum dish=33.8050 grams.
Platinum dish ....=33.7550 "
= 0.0500 (Total sulphates) "
($MgSO_4, K_2SO_4, Na_2SO_4$).

Crucible + $Mg_2P_2O_7$ = 10.729 grams.
Crucible = 10.721 "
0.008 "

$Mg_2P_2O_7$=0.008 gms.=0.008 $MgSO_4 \times 2$ = 0.0176 ($MgSO_4$) "
0.0324 ($K_2SO_4+Na_2SO_4$) "
K_2PtCl_6 = 0.0232 = 0.0082 $K_2SO_4 \times 2$ = 0.0164 (K_2SO_4) "
0.0160 (Na_2SO_4) "
0.0176 $MgSO_4$ = 0.0058 MgO and is added to MgO in (7)

MgO from (7) 0.004
MgO from (8) 0.0058
0.0098

$$\frac{0.0098 \times 2.5 \times 100}{2} = 1.22 \text{ per cent. MgO.}$$

0.0164 K_2SO_4 = 0.0088 K_2O then $\frac{0.0088 \times 2.5 \times 100}{2}$ = 1.10 per cent. K_2O.

0.0160 Na_2SO_4 = 0.0069 Na_2O then $\frac{0.0069 \times 2.5 \times 100}{2}$ = 0.86 per cent. Na_2O.

(SO_3).Crucible + $BaSO_4$ .... 10.729 grams.
Crucible .... 10.721 "
$BaSO_4$ = 0.008 "
SO_3 = 0.0027 "

$$\frac{0.0027 \times 5 \times 100}{2} = 0.67 \text{ per cent. } SO_3.$$

RESUMÉ.

SiO_2	24.20	per cent.
Al_2O_3	6.22	"
Fe_2O_3	3.00	"
CaO	62.67	"
MgO	1.22	"
K_2O	1.10	"
Na_2O	0.86	"
SO_3	0.67	"
Total	99.94	"

The following well known brands of Portland cements were analyzed in my laboratory by above method.

	Burham's.	Dyckerhoff's.	Saylor's.
SiO_2	21.70 per cent.	19.05 per cent.	21.25 per cent.
Al_2O_3	6.82 "	7.90 "	4.21 "
Fe_2O_3	2.37 "	4.48 "	8.25 "
CaO	62.26 "	63.62 "	61.25 "
MgO	1.48 "	1.87 "	1.50 "
K_2O	1.84 "	0.88 "	1.01 "
Na_2O	0.98 "	0.36 "	0.99 "
SO_3	1.20 "	0.94 "	1.38 "
CO_2	1.30 "	... "	... "
	99.95 "	100.00 "	99.84 "

In some cements quartz is a constituent in amounts varying from five-tenths to six per cent. It can be separated from combined silica by the method of Fresenius.[1]

Where carbonic acid has been indicated by the qualitative analysis the quantitative analysis, for this constituent, should be made upon at least eight grams of the cement.

The carbonic acid rarely reaches one per cent., and while it is generally absent in well burned cements, it is by no means an uncommon constituent to the amount of 0.15–0.30 per cent., as the following table of analyses of German cements will show:[2]

	1	2	3	4	5	6	7	8
CaO	61.99	62.89	63.71	63.27	65.59	59.96	64.51	60.81
SiO_2	23.69	22.80	25.37	19.80	22.85	23.70	22.38	22.63
Fe_2O_3	2.71	3.40	3.14	3.22	2.76	3.15	2.24	2.42
Al_2O_3	8.29	7.70	4.31	6.73	5.51	8.20	9.45	7.06
MgO	0.47	1.20	1.25	2.02	1.24	1.00	...	2.89
Alkalies	0.95	1.30	0.84	1.48	0.92	1.05	...	2.83
SO_3	0.69	0.71	0.87	1.08	1.69	0.88	1.44	0.47
CO_2	0.27	...	...	0.23	...	0.26	...	0.33
Insoluble	0.44	...	...	1.38	...	0.80	...	...

The Mechanical Testing.

The method recommended for use in this country by the American Society of Civil Engineers is as follows:

(1) Determination of fineness.

(2) Liability to checking or cracking.

(3) Tensile strength.

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[1] *Quant. Chem. Anal.*, p. 259.

[2] *Der Portland-cement und seine Anwendungen im Bauwesen*, Berlin, 1892, p. 18.

Fineness.—Tests should be made upon cements that have passed through a No. 100 sieve (10,000 meshes to the square inch), made of No. 40 wire, Stubb's wire gauge. The finer the cement the more sand it will unite with and the greater its value.

Liability to Checking or Cracking.—Make two cakes of neat cement two or three inches in diameter, about one-half inch thick, with thin edges. Note the time in minutes that these cakes, when mixed with water to the consistency of a stiff, plastic mortar, take to set hard enough to stand the wire test recommended by General Gillmore, one-twelfth inch diameter wire loaded with one-fourth pound, and one twenty-fourth inch diameter wire loaded with one pound.

One of these cakes, when hard enough, should be put in water and examined from day to day to see if it becomes contorted or if cracks show themselves at the edges, such contortions or cracks indicating that the cement is unfit for use at that time. In some cases the tendency to crack, if caused by too much lime, will disappear with age. The remaining crack should be kept in the air and its color observed, which, for a good cement, should be uniform throughout.

Tensile Strength.—One part of the cement mixed with three parts of sand[1] for the seven days and upward test, in addition to the trials of the neat cement. The proportions of cement, sand and water should be carefully determined by weight, the sand and cement mixed dry, and all the water added at once. The mixing must be rapid and thorough, and the mortar, which should be stiff and plastic, should be firmly pressed into the molds with the trowel without ramming and struck off level, the molds in each instance, while being charged and manipulated, to be laid directly on glass, slate or other non-absorbent material. The molding must be completed before incipient setting begins. As soon as the briquettes are hard enough to bear it, they should be taken from the molds and kept covered with a damp cloth until they are immersed. For the sake of uniformity, the briquettes, both of neat cement and those con-

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[1] White crushed quartz, which passes through a No. 20 sieve, but remains upon a No. 30 sieve, is standard.

taining sand, should be immersed in water at the end of twenty-four hours, except in the case of one day tests. Ordinary clean water having a temperature between 60° F. and 70° F. should be used for the water of mixture and immersion of sample. The proportion of water required is approximately as follows:

For briquettes of neat cement, about twenty-five per cent.

For briquettes of one part cement, one part sand, about fifteen per cent. of total weight of cement and sand.

For briquettes one part cement, three parts sand, about twelve per cent. of total weight of cement and sand.

The object is to produce the plasticity of plasterer's stiff cement.

An average of five briquettes may be made for each test, only those breaking at the smallest section to be taken. The briquettes should always be put in the testing machine and broken immediately after being taken out of the water, and the temperature of the briquettes and of the testing room should be constant between 60° F. and 70° F.

The following table shows the average minimum and maximum tensile strength per square inch which some good cements have attained. Within the limits given the value of a cement varies closely with the tensile strength when tested with the full dose of sand.

AMERICAN AND FOREIGN PORTLAND CEMENTS.—NEAT.

One day, (1 hour, or until set, in air, the rest of the 24 hours in water,) from 100 to 140 pounds per square inch.

One week, (1 day in air, 6 days in water), from 250 to 550 pounds per square inch.

One month, 28 days, (1 day in air, 27 days in water), from 350 to 700 pounds per square inch.

One year, (1 day in air, the remainder in water), from 450 to 800 pounds per square inch.

AMERICAN AND FOREIGN PORTLAND CEMENTS.—1 PART OF CEMENT TO 3 PARTS OF SAND.

One week, (1 day in air, 6 days in water), from 80 to 125 pounds per square inch.

One month, 28 days, (1 day in air, 27 days in water), from 100 to 200 pounds per square inch.

One year, (1 day in air, the remainder in water), from 200 to 350 pounds[1] per square inch.

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[1] In regard to modification of these conditions required for tensile strength, consult *Trans. Amer. Soc. of Civil Engineers*, August, 1891, p. 285.

The machines for determining the tensile strength of Portland cements in use in this country are the "Fairbanks," Fig. 52, the "Riehle," Fig. 53 and the Olsen.

The Fairbanks machine is automatic and is operated as follows:

Hang the cup on the end of the beam; see that the poise is at the zero mark and balance the beam by turning the ball. Place the shot in the hopper. Place the briquette in the clamps and adjust the hand wheel so that the graduated beam will be inclined upward about 45°. Open the automatic valve so as to allow the shot to run slowly. When the specimen breaks the beam drops and closes the valve through which the shot has been pouring. Remove the cup with the shot in it and hang the counterpoise weight in its place. Hang the cup on the hook under the large balance ball and proceed to weigh the shot, using the poise on the graduated beam, and the weights on the counterpoise weight. The result will show the number of pounds required to break the specimen.

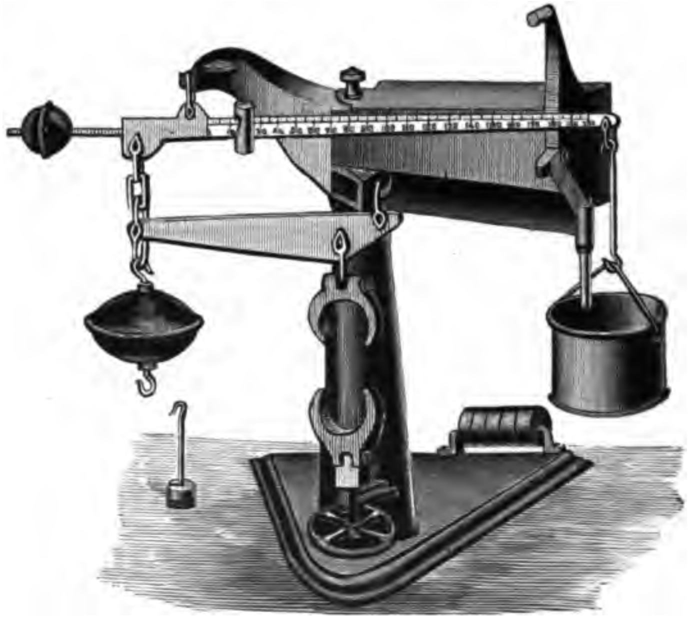

Fig. 52.

The "Riehle," while not automatic, is accurate, and responds to differences as slight as one pound in 2,000. The distinctive features are:

(*a*) The poise moves quietly and smoothly on the weighing beam.

(*b*) The weighing beam is long and the marks not too close together. The slightest movement of the beam is promptly and plainly observed by the motion of the indicator.

(*c*) The levers are tested and sealed to U. S. standard weight.

(*d*) The arrangement of the "grips" to hold the briquette is such that they are always swung from pins, thus giving the test upon the cement when the briquette is on a dead straight line.

Fig. 53.

Directions for Testing Portland Cement According to the Official German Rules.[1]—The quality of a mortar made with cement depends not only on the strength of the cement itself, but also

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[1] *Portland Cement*, by Gustav Grawitz.

on the degree of sub-division of the same. It is therefore necessary to make the tests both with neat cement and with a mixture of the same with "standard sand." This latter as used at the Royal Testing Station at Berlin, is produced by washing and drying quartz sand, which must be clean as possible, and afterwards be sifted through a sieve of sixty meshes per square centimeter (387 meshes per square inch), by which process the coarsest particles are separated. The sand is again sifted through a sieve having 120 meshes to the square centimeter (774 meshes per square inch). The residue remaining in this sieve is the standard sand for experiments, the coarsest and finest particles having been eliminated. It is absolutely necessary in order to obtain uniform results to use only the "standard sand," as the size of the grain has a material influence on the results of the testing. The sand must be clean and dry, and all earthy and other substances previously removed by washing.

Preparation of Briquettes of Neat Portland Cement.—Upon a slab of metal or marble are laid five sheets of filtering paper, which have been previously saturated with water, and upon these are placed five brass molds (Fig. 54) thoroughly cleaned and moistened with water. One thousand grams of cement and 250 grams of water must be thoroughly mixed, well worked up, and when the resulting mass has been rendered perfectly homogeneous, it is poured into the molds. The latter must be gently tapped by means of a wooden hammer with equal force on both sides during ten to fifteen minutes to insure the escape of confined globules of air. The molds must be carefully filled up until the mass becomes plastic, the superfluous mortar is then struck off, and the mold carefully withdrawn. The samples, after remaining twenty-four hours exposed to the air, at a temperature of about 60° F., must be immersed in water having the same temperature, and care must be taken that they remain covered with water until the time arrives for breaking them. In order to obtain a proper average at least ten briquettes should be prepared for every examination.

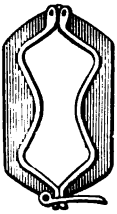

Fig 54.

Preparation of Briquettes from a Mixture of Portland Cement and Standard Sand.—Place the molds on metal as described in preparation of neat cement briquettes. The quantities (by weight) specified of cement and sand are thoroughly mixed and to this is added the requisite quantity of water. The whole mass is then worked up with a trowel or spatula until it becomes uni-

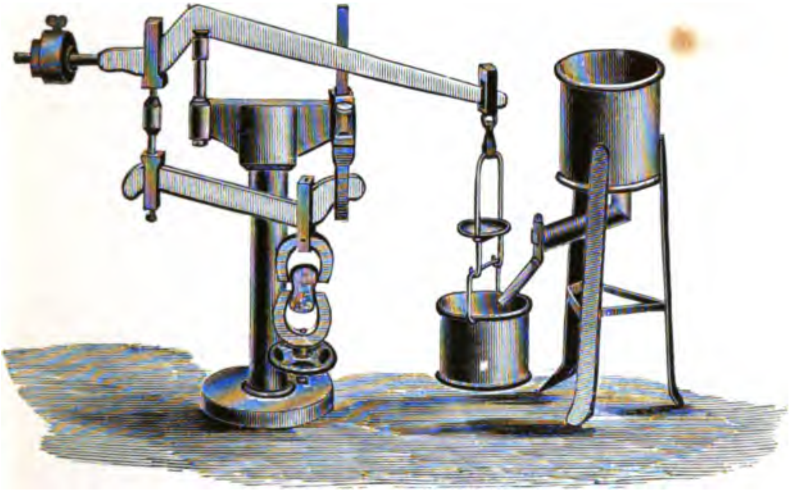

Fig. 55.

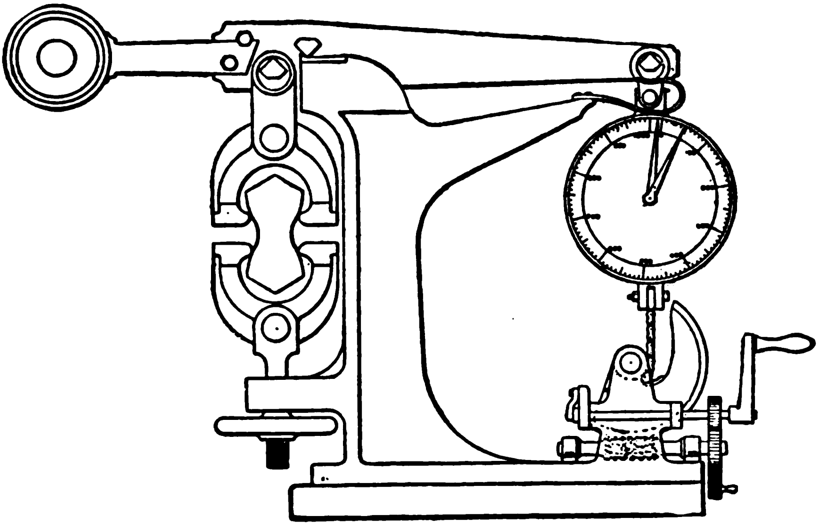

Fig. 56.

form. In this manner is obtained a very stiff mortar. The molds are filled and mortar heaped up. The latter is then beaten into the molds with an iron trowel, at first lightly, and afterwards more heavily, until it becomes elastic and water appears on the surface. The superfluous morter is then scraped off with a knife and by means of the same the surface is leveled. The further treatment of these briquettes is the same as for neat cement briquettes. The average of ten breaking weights furnishes the strength of the mortar tested.

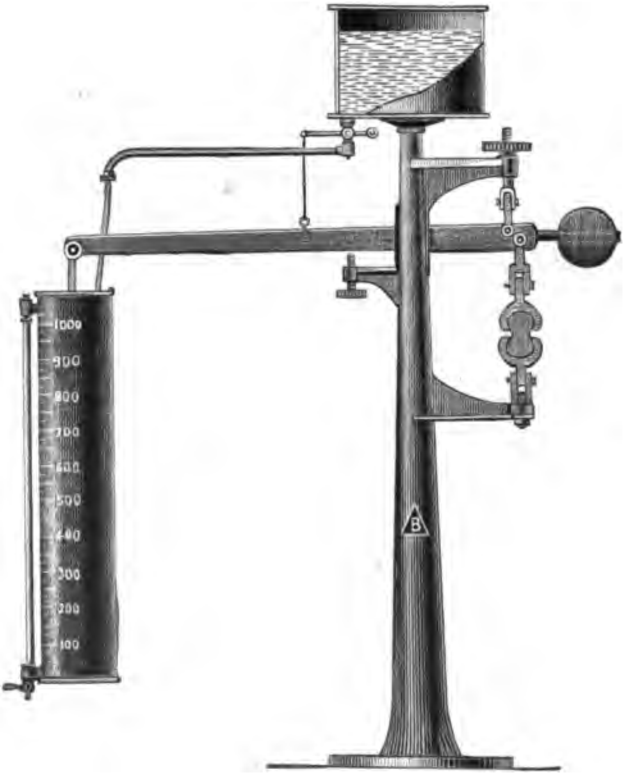

Fig. 57.

The machine in general use in Germany for determining the tensile strength of cements is the Michaelis (Fig. 55), and from this is derived, with modifications, the "Reid and Bailey" machine in use in England, and the "Fairbanks" previously described.

No standard specifications for the testing of Portland cement are required in Great Britain, the determination of fineness, tensile strength and variations in volume, being considered sufficient to determine the value of a cement. The machines for tensile strength are the "Faija," (Fig. 56), the "Reid and Bailey," (Figs. 57 and 58), or the "Grant," similar to the Riehle, and described in *Proceedings* of the Institution of Civil Engineers, **62**, 113. The "Reid and Bailey" is essentially the "Michaelis" (Fig. 55), excepting that water is used instead of fine shot for the breaking power.

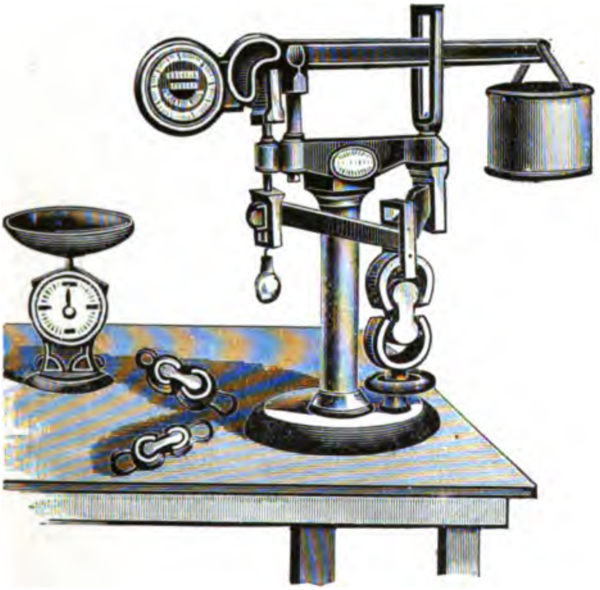

Fig. 58.

It is readily seen that the "Faija" and "Grant" machines, not being automatic, require the application of the power at a certain uniform speed to obtain comparable results, since a difference of twenty-five per cent. of tensile strength may be obtained by applying the strain very quickly or very slowly.[1]

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[1] Proceedings of the Institution of Civil Engineers, 75, 225, 226.

Faija has determined this variation with extreme care, the results being indicated in the curve shown in Fig. 59. To overcome these variations a uniform speed of 400 pounds per minute has been accepted as the standard.

Not only are comparable methods required in the use of the machines to obtain uniform results, but the briquettes must also be constructed under similar conditions.

It is manifestly unjust to compare the tensile strength of two cements (even when the briquettes are broken upon the same machine) unless the briquettes have the same weight of water for mixing; the same pressure with the trowel when being formed in the molds, and the same length of time of exposure under water before submitting the briquettes to the tensile strain.

For instance: Comparing tests made upon the Dyckerhoff Portland cement by Dr. Böhme, Director of the Royal Commission for testing building material, at Berlin, and by E. J. DeSmedt, General Inspector Engineering Department, District of Columbia, we find that the German method gives a much higher tensile strength than the method in use in this country.

DR. BÖHME.

Age of briquettes.	Average tensile strength per square inch.	Number of tests.
7 days	767 pounds	10
28 "	895 "	10

E. J. DESMEDT, C. E.

Age of briquettes.	Average tensile strength per square inch.
5 days	250 pounds.
30 "	700 "

Showing:

109 pounds increase per day (7 days), Dr. Böhme.
59 " " " " (5 days), DeSmedt.

or over 100 per cent, difference upon the same cement on the seven days test.

These variations are undoubtedly due principally to the different pressures upon the cement during the making of the briquettes, and to overcome difficulties of this nature the Vereins deutsche Portland Cement Fabrikanten have modified the rules

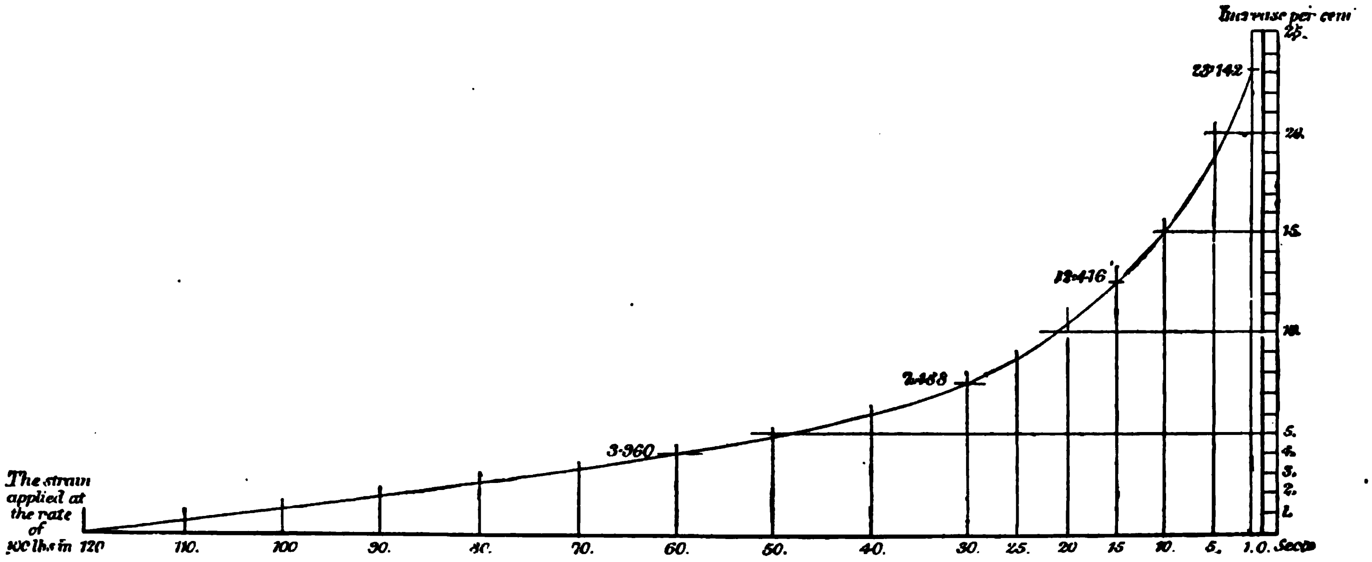
Increase per cent
25.
20.
15.
10.
5.
4.
3.
2.
1.
23·142
12·416
7·488
3·960
The strain applied at the rate of 100 lbs in
120
110.
100
90.
80.
70.
60.
50.
40.
30.
25.
20
15
10.
5.
1.0.
Secs

Fig. 59.

in the construction of the briquettes so that two methods are acceptable :

First, the normal method, above given, with the trowel, etc. (" Handarbeit ".)

Second, the use of the Böhme-Hammer apparatus or " machine method," by which the cement in briquette form (after

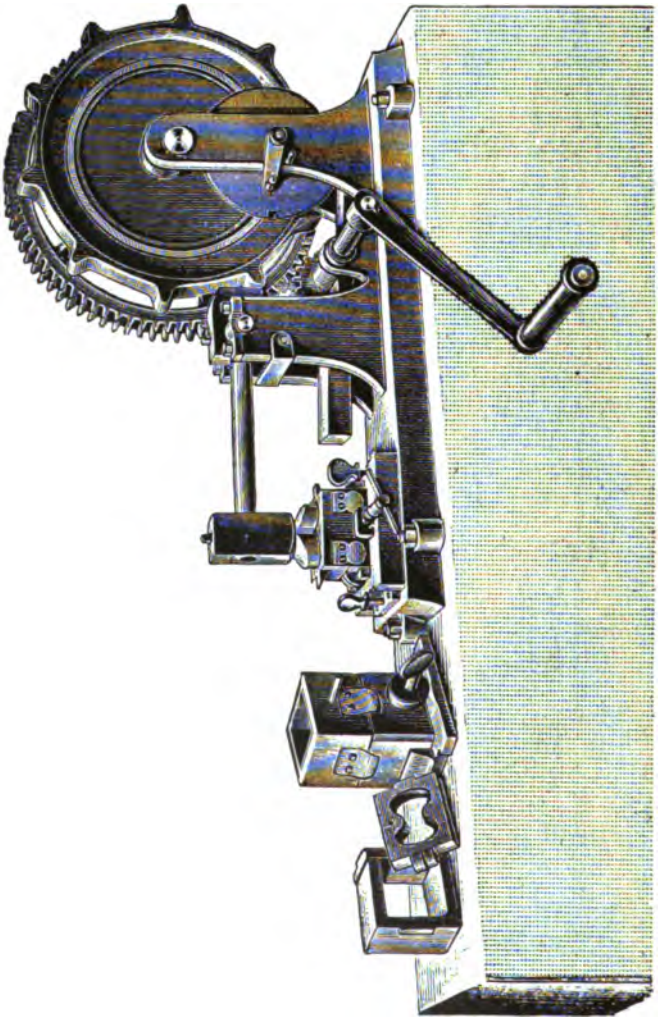

Fig. 60.

mixing with proper amount of water), is submitted to a pressure of 150 blows from a hammer weighing two kilos (Fig. 60). The briquette of cement is then removed from the mold and treated for tensile strength as usual.

This subject is receiving considerable attention at the present moment, the evident purpose being to render the tests of tensile strength as uniform as possible by making the working of the apparatus automatic and the production of cement briquettes with the least possible variation in the pressure in the molds.

In this case, no matter how careful the experimenter may be, the "personal equation" enters largely into the results of testing hand-made briquettes, for which reason the manufacture of the briquettes should be as automatic as possible. In no other way can results obtained by different experimenters be compared.

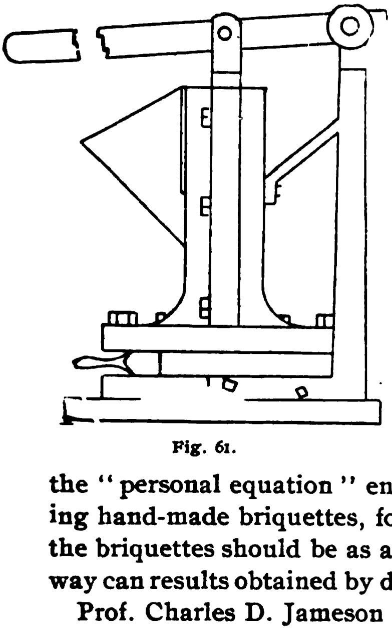

Fig. 61.

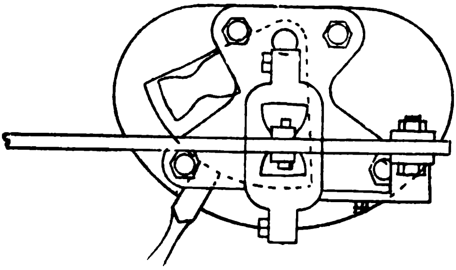

Fig. 62.

Prof. Charles D. Jameson describes an apparatus for this purpose (Figs. 61 and 62).[1]

The method of operating is as follows: The lever being raised so that the lower end of the piston or main plunger is above the hole in the side of the cylinder communicating with the hopper, cement is put in the hopper and pushed down into the cylinder. The molding plate is pushed against one of the stops, so that

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[1] Transactions of the American Society of Civil Engineers, 15, 302.

Results of Tensile Tests on the Same Sample of Cement.

(Compiled by Prof. J. M. Porter, Lafayette College.)

MADE BY DIFFERENT PERSONS IN ACCORDANCE WITH THEIR UNDERSTANDING OF THE METHOD PROPOSED BY THE COMMITTEE OF AMERICAN SOCIETY OF CIVIL ENGINEERS.

(One part cement to three parts sand. Average, seven days).

Laboratory.			Briquettes broke at: in pounds.							Manipulation of briquettes.					
No.	Name.	Report Dated.	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	No. 6.	Aver.	Water per ct.	Molds.	Struck off.	Plc'd in water.	Water-bath.	Machine.
1	R. W. Hildreth & Co., New York.	1895 10/22	68	72	74	78	82	..	**75**	12.0	Single.	Both sides.	Flat-wise.	Still.	Riehle, driven by hand.
2	Washington University, St. Louis, Mo., Prof. J. B. Johnson.	10/19	77	94	108	110	122	..	**102**	Not known	Gang.	One side.	Flat-wise.	Still.	Fairbanks Johnson's clip.
3	City of Easton, Pa., H. R. Fehr, City Engineer.	10/11	106	112	123	..	..	..	**114**	10.4	Single.	Both sides.	Edge-wise.	Still.	Fairbanks.
4	Columbia College, New York, Prof. W. H. Burr.	10/18	125 126	126 132	130 138	137 144	140 150	144 153	**133** **140**	10.0 8.0	Single. Single.	One side.	Flat-wise.	Still. Still.	Riehle, driven by hand.
5	Chas. F. McKenna, New York.	9/24	148	150	151	155	160	..	**153**	12.0	Single.	One side.	Flat-wise.	Still.	Riehle-McKenna power. Rubber clip.
6	Cornell University, Ithaca, N. Y.,	10/23	155	160	164	166	172	..	**163**	Not given.	Single.	Both sides.	Edge-wise.	Running.	Lever with water bucket.
7	Lafayette College, Easton, Pa., J. M. Porter.	9/24	171	177	177	178	179	..	**176**	11.0	Gang.	Both sides.	Edge-wise.	Running.	Olsen-Porter. Porter clip.
8	Clifford Richardson, Washington D. C.	10/7	220	224	226	228	228	..	**225**	10.0	Single.	Both sides.	Flat-wise.	Still.	Riehle, driven by hand. Rubber clips.
9	Booth, Garret & Blair, Philadelphia, Pa.	9/24	240	246	249	250	252	..	**247**	12.0	Single.	Both sides.	Edge-wise.	Still.	Olsen power. Rubber clip.
	Average.								**153**	10.8	77.8 % Single. 22.2 % Gang.	66⅔ % Both sides. 33⅓ % One side.	55.5 % Flat. 44.5 % Edge.	22.2 % Running. 77.8 % Still.	66⅔ % power. 33⅓ % hand.

NOTE.—All molds and sand are American Society of Civil Engineers standard.

one of the openings is beneath the bore of the cylinder. The long lever is forced down, causing the plunger to force the cement into the opening of the molding plate. After this, the molding plate is swung against the other stop, cutting off the briquette, placing it over the plungers, throwing the other opening in the molding plate directly beneath the cylinder. The smaller lever is lifted, raising the plunger, and forces the briquette out of the mold, after which it is removed. The plunger is then pressed down, the main lever also, the molding plate swung back to the first position, the other plunger lever lifted, and another briquette is ready to be taken away, and so on.

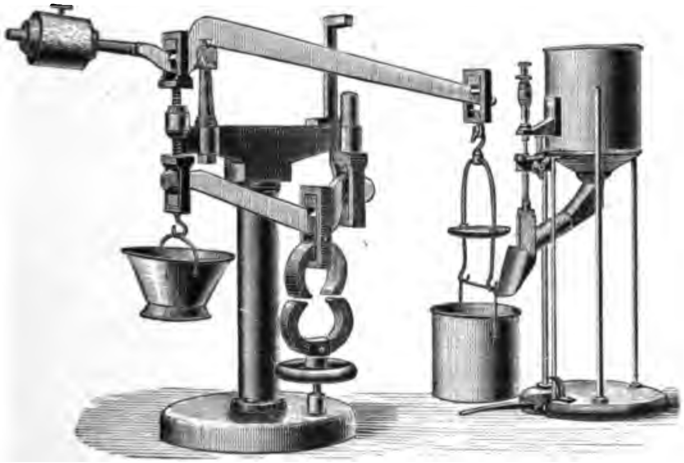

Fig. 63.

After making three briquettes, the main lever is lifted and more cement placed in the cylinder. The machine is best operated by two men, one to feed and operate the long lever, and the other to swing the molding plate, remove the briquettes and lower the plungers. The pressure on the briquette is 175 pounds per square inch.

The conditions required in France for a good cement are: [1]

First. Analysis to determine the chemical composition.

Second. The determination of density.

Third. The determination of fineness.

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[1] E. Candlot, *Ciments et Chaux Hydrauliques*, Paris, Baudry & Co. 1891.

Fourth. The determination of tensile strength.

Fifth. The determination of crushing strength.

Sixth. The determination of variations in volume.

The tensile strength is determined by the use of a Michaelis machine, Fig. 63, or the use of a Buignet apparatus, Fig. 64, this latter being upon an entirely different principle than any yet in use, and is thus described by the designer, M. Buignet, Conductor des ponts et chaussées au Havre:

It is composed of a basin *A* and frame *B*.

The basin *A*, filled with mercury and water, closes up by a diaphragm of rubber covered with a metallic disk, and is in direct communication with:

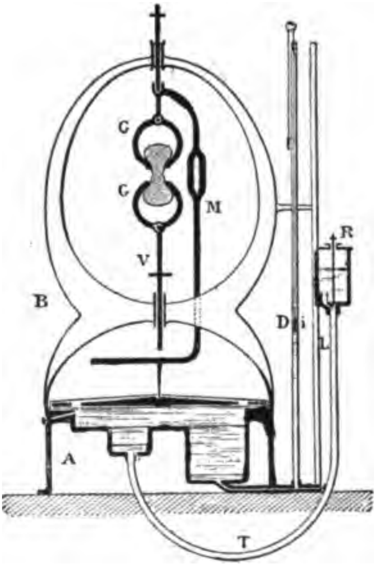


Fig. 64.

(*a*) Manometrique tube *D*.

(*b*) With a movable reservoir *R*, filled with mercury, by means of a thick rubber tube *T*.

The grips *G G*, in which are to be placed the briquettes to be tested, are fastened, one to the frame *B* by the support *V*, the other to the support *M*, which rests upon the center of the metallic disk over *A*. It is operated as follows: The briquettes are placed in the grips *G G*, and the support *V* moved up or down until equipoise is established, and then firmly secured by a crank in frame *B*.

The support *M* is adjusted until the point at its lower end just touches the metallic disk in *A*.

By gradually lowering the reservoir *R* an upward pressure is given to the metallic disk in *A*, which is transferred to the support *M*, until when sufficient pressure is exerted the briquette is broken. The moment rupture of the briquette takes place, the pressure required to do this is indicated by the float *i* in the manometer tube *D*.

By a comparison of the various machines used in Germany, England, France, and the United States, we find practically but two in general use: the "Michaelis" and the "Grant." While nearly all engineers require cements to be subjected to the tensile strength test, in fact relying more upon this one test than any of the others, it might be well to include here the opinion of H. Le Chatelier, professor at the École des Mines, Paris, France, as given in a paper presented at the last meeting of the American Institute of Mining Engineers, August, 1893, entitled "Tests of Hydraulic Materials," p. 44.

"The method of tension is at present most widely used, but the preference for it is not well founded. Here, as in rupture by bending, only the *surface* of the briquettes acts in a really useful way, and its inevitable irregularities and alterations so greatly affect the precision of the results that they can in no case be trusted nearer than about twenty per cent.

"This preponderant influence of the superficial parts was first shown by the fact that the resistance of briquettes of different sizes increases, not with the section, but, on the contrary, with the perimeter. Finally, M. Durand-Claye has shown that the interior of a briquette may be removed without notably diminishing its resistance to rupture by tension, and has given a complete theoretical explanation of the phenomena which seemed at first sight paradoxical."

The Crushing Test.

This test is not official in this country and is seldom required by our engineers, who, however, have confined their experiments in this direction mainly to crushing tests of concrete, formed by mixing Portland cement, sand, and broken stone.

Tests upon cubes of neat cement and of mortar composed of one part cement and three of standard sand, are generally included in reports given upon the examination of cements in Europe, the ratio being that the crushing strength is about ten times greater than the tensile strength.

Thus, a cement of good quality should show the following resistance per square centimeter:

TENSILE STRENGTH.

	7 days.	28 days.
Neat cement	25 kilos.	35 kilos.
1 part cement 3 parts sand	10 "	18 "

CRUSHING STRENGTH.

	7 days.	28 days.
Neat cement	250 kilos.	350 kilos,
1 part cement 3 parts sand	100 "	180 "

To convert kilos per square centimeter to pounds per square inch, the equivalents used are: one kilo = 2.204 pounds English; 6.451 square centimeters = one square inch, English.

The hydraulic presses made use of for this purpose, a few years since, gave very discordant results, as it was impossible to distribute the pressure evenly over the surface of the cubes. This has been overcome, and there are now several machines upon the market whose results are comparable, *viz.*:

The "Suchier," Fig. 65, the "Böhme," Fig. 66, the "Tetmajer," as improved by Prof. Amsler-Laffon,[1] the "Brink and Hubner,"[2] the "Riehle," the "Fairbanks," the "Olsen" and the "Bailey."

Variation in volume (expansion or contraction)—The method of Faija,[3] the one generally used for this purpose, is as follows:

---

[1] Consult: *Schweizer Bauzeit*, January 12, 1889.

[2] Description of the "Suchier," "Böhme," and "Brink and Huber" machines will be found in *Der Portland Cement und seine Anwendungen im Bauwesen*, Berlin, 1892.

[3] The determination of liability to "checking" or "cracking" (variation in volume) in Portland cements as recommended by *American Society Civil Engineers*, is not as complete as Faija's method. See *J. Am. Chem. Soc.*, 15, 184.

Three pats should be made on pieces of glass or other non-porous substance, and their behavior watched under the following conditions:

Pat No. 1 may be left in the air, and No. 2 should be put in water as soon as it is set hard.

Fig. 65.

Pat No. 3 should be treated in the apparatus for determining the soundness of cement. The apparatus consists of a covered vessel in which water is maintained at an even temperature of 110° C.; the space above the water is therefore filled with the vapor rising therefrom, and is at a temperature of about 100° C. Immediately the pat is gauged, it should be placed on a rack in the upper part of the vessel, and in five or six hours it may be placed in the warm water and left therein for nineteen or twenty hours. If, at the end of that period, the pat is still fast to the glass and shows no signs of blowing, the cement may be considered perfectly sound; should, however, any signs of blowing appear, the cement should be laid out in a thin layer for a day or two, and a second pat made and treated in the same manner, as the blowing tendency may only be due to the extreme newness of the cement.

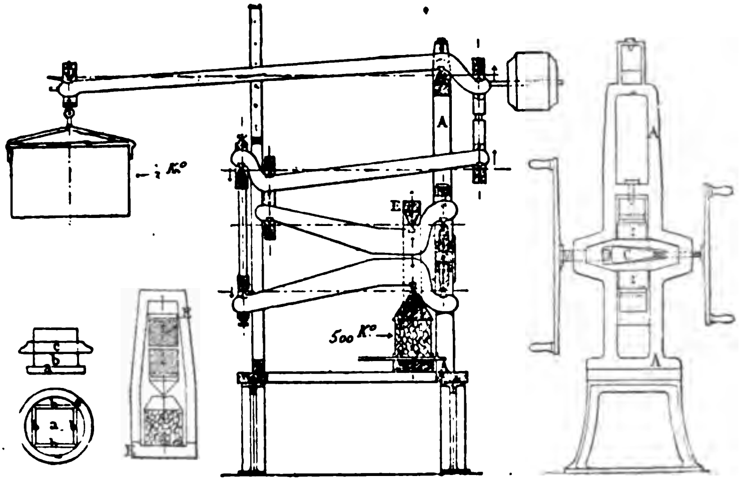


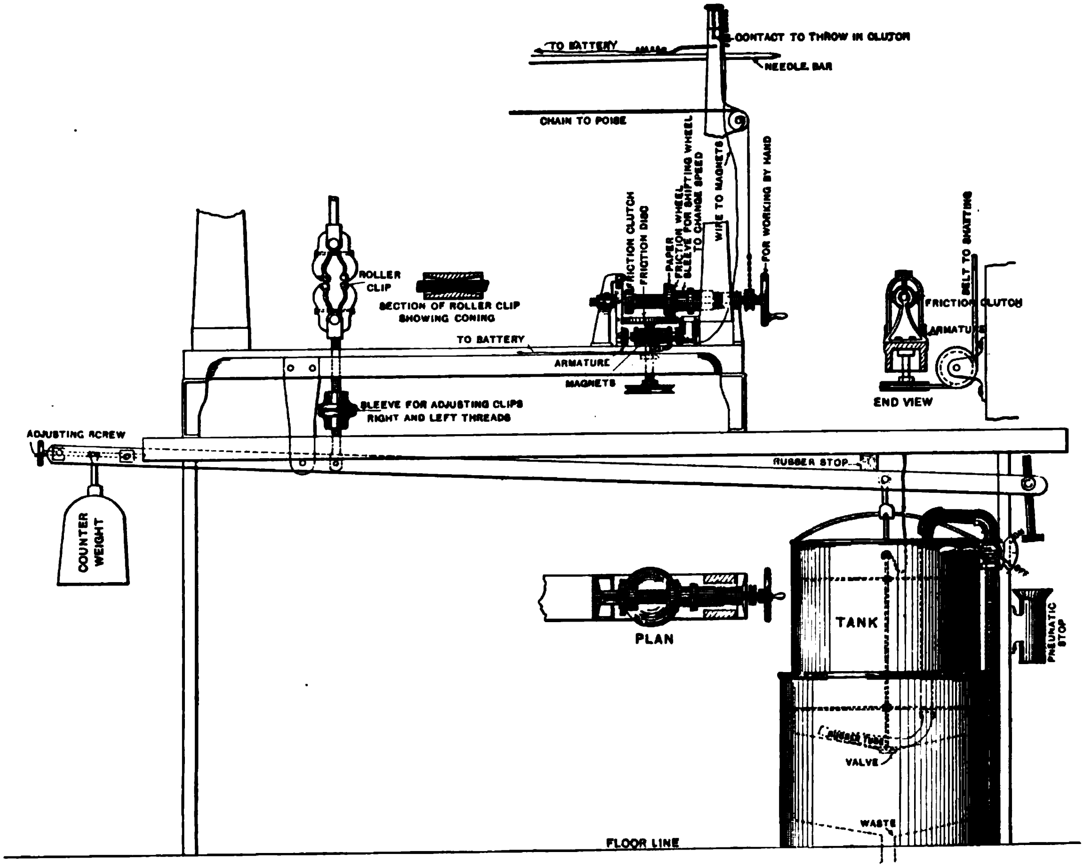
Fig. 66.

If pat No. 3 shows the cement to be unsound, pats Nos. 1 and 2 will eventually prove it, but it may be weeks or even months before they develop the characteristics. If pat No. 2 blows, it may be because it was put in the water before it was set. A cement is considered set hard when it can no longer be marked by the pressure of the thumb nail.

An Automatic Cement Testing Machine.

To promote convenience and rapidity and secure uniformity, regularity, and a standard method of work as free as possible from the irregularities coming under the head of "personal equation," Prof. J. M. Porter has devised the adjustable automatic loading and balancing attachments which are illustrated by the accompanying elevation and details of the special mechanism added to the 2,000-pound Olsen machine of standard pattern. Fig. 67. The load is applied by filling with water a tank suspended from the long arm of a 15 to 1 lever, the connection of which has a pin bearing on a cylindrical surface which rests on the adjustment screw of the lower grip. Neither the tank nor its contents are weighed, but the exact rate of loading per minute is accurately known from previous tests. Water is admitted to the tank from a large reservoir on the roof, where a practically constant height of surface level is maintained, so that there is no sensible variation of pressure in the stream admitted through a carefully fitted gate valve. The position of this valve at "open," "closed," and all intermediate points is shown by an index attached to the stem and registering on a dial marked off with the number of pounds per minute applied to specimen as determined and verified by previous experiments.

When the specimen breaks, the load lever drops and permits the load tank to fall a few inches, so that the chain is brought into tension and arrests the descent of the valve before its seat stops descending. Thus the bottom of the tank is opened and the contents quickly escape into the hopper of the receiving case, and are carried off through the waste to the sewer. The actual load can be applied at from zero to eighty pounds per minute, thus giving an increase strain of zero to 1,200 pounds per minute on the specimen. A small electric motor is belt-connected to the pulley that continuously drives a friction disk and its engaged wheel. The wheel is feathered to a sleeve that runs loose on its shaft, and carries a coned clutch that is nominally disengaged from its cone, which is feathered to shaft, and can be moved slightly longitudinally on the shaft into contact with the wheel by the action of a lever.



TO BATTERY
CONTACT TO THROW IN CLUTCH
NEEDLE BAR
CHAIN TO POISE
FRICTION CLUTCH
FRICTION DISC
PAPER
FRICTION WHEEL
SLEEVE FOR SHIFTING WHEEL TO CHANGE SPEED
WIRE TO MAGNETS
FOR WORKING BY HAND
BELT TO SHAFTING
FRICTION CLUTCH
ARMATURE
END VIEW
ROLLER CLIP
SECTION OF ROLLER CLIP SHOWING CONING
TO BATTERY
ARMATURE
MAGNETS
SLEEVE FOR ADJUSTING CLIPS RIGHT AND LEFT THREADS
ADJUSTING SCREW
RUBBER STOP
COUNTER WEIGHT
PLAN
TANK
PNEUMATIC STOP
VALVE
WASTE
FLOOR LINE

Fig. 67.

When the scale beam rises, it makes a contact which completes the electric circuit and sends a current through the electromagnet and causes it to attract its armature (here shown not in contact), which moves to the right about a pivot a sufficient distance to make the friction clutch with the coned wheel and drive shaft. This shaft in turn operates the sprocket wheel and chain, which draw the weight out on the scale beam until the latter falls, and breaking the electric circuit, releases the armature and allows the friction clutch to disengage. By turning the capstan-head nut, the friction wheel is set at a greater or less distance from the center of the disk, and the chain is overhauled faster or slower accordingly. The arrangement was constructed in the college laboratory and is positive and simple. It does not get out of order and is considered accurate and satisfactory, and to enable more rapid and better comparable tests to be made more than twenty specimens per hour have been broken by its use.

Resumé: The determination of the value of Portland cement therefore requires the following tests:

First. Chemical analysis.

Second. Determination of fineness.

Third. Determination of tensile strength, including the use of automatic briquette machines as well as an apparatus for mixing the cement with water, as "Faija" mixing machine.

Fourth. Determination of crushing strength.

Fifth. Determination of variation of volume.

References: J. Am. Chem. Soc., 16, 382–386, contains an index, arranged by the writer, of the literature relating to Portland cement, from 1870 to 1893.

XXVIII.

Determination of Nickel.

The principles involved in the processes are the following:[1]

First. The iron is precipitated as ferric phosphate in cold, strong acetic acid solution, under which condition it precipitates perfectly free from nickel, although retaining a small amount of copper.

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[1] E. D. Campbell: *J. Am Chem. Soc.*, 17, 125.

Second. The copper is separated from manganese and nickel in hydrochloric acid solution by means of granulated lead.

Third. The manganese and lead, which displaced the copper, are separated from the nickel by means of cold ammoniacal solution of sodium phosphate.

Fourth. The nickel is determined in the ammoniacal filtrate from the phosphate of manganese and lead, by titration with standard potassium cyanide, or by electrolytic deposition.

In case the nickel is accompanied by cobalt the latter metal remains with the nickel and may be separated from it by any of the well-known methods after dissolving off the electrolytically deposited nickel.

The two methods described below are identical up to the point where a portion of the filtrate from the phosphates of manganese and lead is taken. The description of that part of the methods common to both will be first given, and then the two ways of treating the above filtrate for the final determination of nickel will be added.

Take 2.2222 grams of nickel-steel, place in a 500 cc. graduated flask, add twenty cc. nitric acid, sp. gr. 1.20, and five cc. hydrochloric acid, sp. gr. 1.21. Boil until the solution is clear, which will usually require not more than from five to ten minutes. Remove from the plate and add 155 cc. sodium phosphate solution. If a slight precipitate should form which does not dissolve upon shaking, add carefully a few drops of hydrochloric acid until the solution clears up. Add twenty-five cc. acetic acid, sp. gr. 1.04, then 100 cc. sodium acetate solution, shake, dilute with water to 502.5 cc., shake again, and allow to stand fifteen minutes. Filter through a dry twenty-five cc. filter, catching the filtrate in a dry beaker. As soon as enough of the filtrate has run through, which requires about ten minutes, draw off with a pipette 250 cc. of the filtrate, transferring to a No. 4 beaker. This will give one-half of the solution, since it was found by experiment that the ferric phosphate from the amount of steel taken occupies two and a half cc. Bring the solution to a boil and add twenty grams potassium hydroxide previously dissolved in forty cc. of water. Boil five minutes, then keep just below boiling-point until the precipitate has settled and the solution is clear. This precipitates copper, manganese, and nickel so completely that the filtrate gives no color with hydrogen sulphide. Filter through asbestos, using a pump, decanting as much of the solution as possible before allowing the precipitate to get upon the filter. Wash with water. Dissolve the precipitate on the filter in a hot solution of six cc. strong hydrochloric acid with an equal volume of water. Wash the filter, using only as much water as is necessary. To the solution in the flask, which should not exceed fifty cc. and should have a temperature of 40° to 50° C., add fifteen grams of granulated lead and agitate at frequent intervals for five or ten minutes. This will completely precipitate the copper, a small amount of lead going into solution. Filter through a small glass wool filter, catching the filtrate in a No. 2 beaker; wash the granulated lead with a small amount of water and boil the solution down until it does not exceed sixty cc. Add ten cc. of sodium phosphate solution, then ammonium hydroxide until a precipitate begins to form, then hydrochloric acid sufficient to clear the solution, cool until cold, and transfer to a cylinder or flask graduated to 111.1 cc. Add five cc. strong ammonium hydroxide, sp. gr. 0.90, dilute to the mark, shake well, and allow to stand fifteen minutes. Filter through a dry nine cm. filter, receiving the filtrate into a dry beaker. Draw off, by means of a pipette, 100 cc. of the filtrate, which is equivalent to one gram of the original steel, and treat by one of the two following methods:

Electrolytic Method.

Transfer the 100 cc. of filtrate, above mentioned, to a large platinum dish having a capacity of about 200 cc. Add twenty-five cc of strong ammonium hydroxide, sp. gr. 0.90, and dilute to 175 cc. Electrolyze for at least four hours, using a current yielding four cc. of electrolytic gas per minute. This strength of current can be easily obtained by connecting three medium-sized cells. The end of the precipitation of the nickel is indicated when a drop of the solution placed in contact with a drop of ammonium sulphide gives no color due to nickel sulphide. When the nickel is completely precipitated, disconnect the battery, wash the nickel thoroughly with water, then finally twice with alcohol, and, after draining off as much as possible, heat for a few minutes in an air bath at 110° C. Cool and weigh. After getting the combined weights of the platinum dish and nickel, dissolve off the latter by warming with five to six cc. of nitric acid (sp. gr. 1.20), then wash the platinum dish by means of water and alcohol, and dry and weigh as before. The difference in the two weighings gives the nickel. It is more satisfactory to weigh the empty dish after the precipitated nickel has been dissolved off than before electrolysis, since in this way a shorter time will elapse between the two weighings and consequently less error will be introduced from variations in atmospheric conditions.

Volumetric Method.

Take 100 cc. of the filtrate from the phosphate of manganese and lead, add hydrochloric acid very carefully until the blue color of the double ammonium nickel chloride disappears, then add ammonium hydroxide, drop by drop, until the blue just reappears, add an excess not exceeding one cc. Dilute to 200 cc., add five cc. of cupric ferrocyanide indicator, and run in standard potassium cyanide until the solution turns from the purple color of the indicator to a perfectly clear light straw-yellow. Subtract from the number of cubic centimeters of potassium cyanide used, the correction for the indicator. The difference gives the amount necessary to convert the nickel into the double cyanide of potassium and nickel. Multiplying this by the factor of the potassium cyanide, expressed in metallic nickel, gives the amount of nickel in one gram of the original sample.

Special Apparatus and Reagents.

Five hundred cc. graduated flask with an additional mark at 502.5 cc.; 250 cc. drop pipette; 100 cc. drop pipette; glass stoppered cylinder or flask graduated to 111.1 cc. The graduated apparatus should be carefully calibrated and compared before using.

Sodium phosphate solution, made by dissolving 200 grams of the ordinary crystallized disodium hydrogen phosphate in 1860 cc. of water. Ten cc. of the solution contain one gram of the crystallized salt, and it requires seventy cc. to precipitate one gram of iron as ferric phosphate.

Sodium acetate solution, made by dissolving 250 grams crystallized sodium acetate in 820 cc. of water. 100 cc. of this solution contain twenty-five grams of sodium acetate, which is a slight excess over that which is necessary to convert the nitric and hydrochloric acids to sodium nitrate aud chloride, with the liberation of the corresponding amount of acetic acid.

Granulated lead is of the same quality as that used in assaying. In size it should be that which passes through a sieve with twenty meshes to the inch, but remains upon a sieve with forty meshes. Before using, the lead should be washed with dilute hydrochloric acid (one part of acid to two parts of water) in order to dissolve any oxide that may be present.

Standard nickel solution. This may be made from chemically pure nickel by dissolving two and a half grams nickel in fifty cc. nitric acid, sp. gr. 1.20, adding an excess of hydrochloric acid, evaporating on a water-bath nearly to dryness, then diluting to one liter. One cc = 0.0045 gram of nickel.

Standard potassium cyanide solution.—Take twelve grams of C. P. potassium cyanide, dissolve in water, dilute to one liter. This must be standardized against a standard nickel solution. Since the presence of ammonium salts interferes somewhat in the titration with potassium cyanide, necessitating the use of a slightly greater amount of potassium cyanide than would be required if there were no ammonium salts present, it is better that the potassium cyanide be standardized under the same conditions as are met in analysis. To standardize the potassium cyanide, take fifteen to twenty cc. of the standard nickel solution, add six cc. of hydrochloric acid, sp. gr. 1.20, ten cc. sodium phosphate solution, ammonium hydroxide until the solution turns blue and then five cc. in excess. Now add hydrochloric acid until the blue color of the double nickel chloride disappears, then ammonium hydroxide until the blue color just reappears, and an excess not exceeding one cc. Dilute to 200 cc., add five cc. cupric ferrocyanide indicator and run in potassium cyanide until the solution changes from the purplish color imparted by the indicator to a perfectly clear light straw-yellow.

Divide the amount of nickel in the standard nickel solution taken, by the number of cubic centimeters of potassium cyanide used, less the correction for the indicator. The result will give the strength of the potassium cyanide expressed in metallic nickel.

Cupric ferrocyanide indicator.—Take two and a half grams of crystallized cupric sulphate, dissolve in twenty-five cc. of water, add to this a solution of ammonium oxalate until the precipitate first formed just redissolves, then dilute to 500 cc. Dissolve two and a half grams of potassium ferrocyanide in 500 cc. of water, then slowly pour this solution into the cupric sulphate solution, stirring constantly during the operation. This will give a deep purplish brown solution of cupric ferrocyanide which may precipitate partially on standing; but the precipitate so formed will be so fine that it will easily remain in suspension for a long time, upon shaking the bottle, thus insuring uniform composition. To find the correction for the indicator take 200 cc. of water, add six to eight drops of ammonium hydroxide, then five cc. of indicator, taken after shaking the bottle well, and then run in potassium cyanide until the characteristic change of color is obtained. Five cc. of cupric ferrocyanide of the above strength require from 0.15 to 0.20 of potassium cyanide, one cc. of which is equivalent to 0.0025 nickel. If a stronger end reaction is desired, ten or even fifteen cc. of the indicator may be used and a suitable correction made.

Repeated analyses of steel have shown that the nickel may be determined, by the volumetric method, within from 0.0003 to 0.0005 gram of the true nickel content, duplicate determinations being made in three hours. The electrolytic method requires three hours to the time the solution is ready for electrolysis.

XXIX.

Analysis of Chimney Gases for Oxygen, Carbon Dioxide, Carbon Monoxide, and Nitrogen.

The determinations usually made are the percentages, by volume, of oxygen, carbon dioxide, carbon monoxide, and nitrogen.

The apparatus used (a modified form of the Elliott) is shown in Fig. 68, and consists of two glass tubes, *ib* and *ah*, the tube *ib* having a capacity of about 125 cc. and is accurately graduated from 0 cc. to 100 cc. in one-tenth cc. At *d* and *e* are three-way

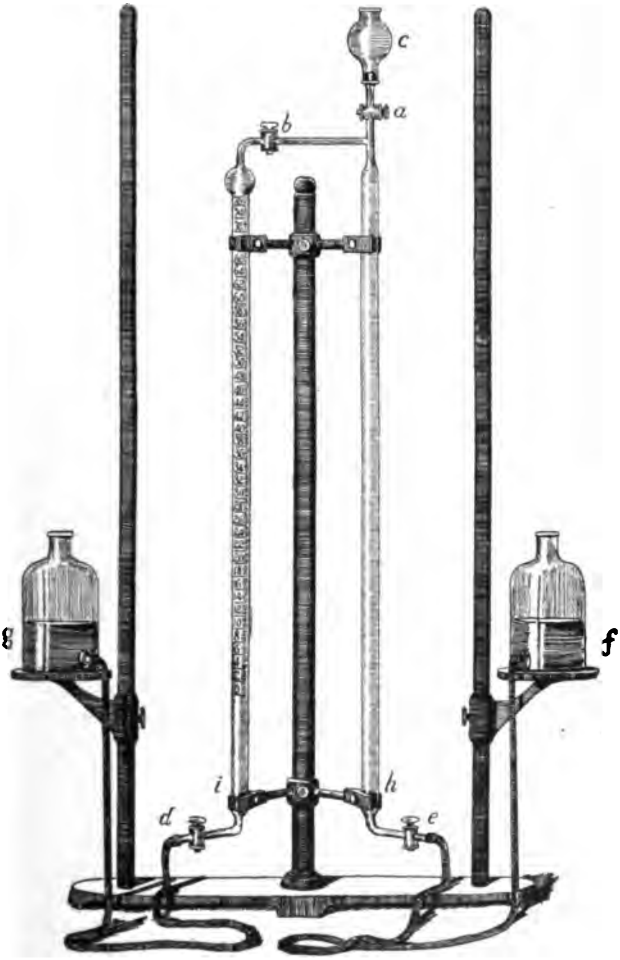

Fig. 68.

glass stopcocks, connected by means of rubber tubing to the water-supply bottles, *f* and *g*.[1] The manipulation of the apparatus is as follows :

Remove the funnel cap *c*, and connect in its place a glass tube of small diameter, but of sufficient length to reach well into the flue from which the gases are to be taken. Open the stop-cocks *a* and *b* and slowly raise *g* and *f* until both tubes are full of water including the glass tube in the flue. It is necessary in this operation to be certain that no air is in the tubes and that the displacement by water is complete. Now gradually lower the bottle *f* whereby the gas is drawn into the tube *ah*. As soon as sufficient gas has been obtained for the analysis, the lower portion of the tube containing water two or three inches above the point *h*, the stop-cock *a* is closed, the small glass tube connecting *a* with the flue removed, and the funnel cap *c* replaced. After allowing the gas to stand in the tube *ah* fifteen minutes to secure it the temperature of the room, and thus insure correct measurements, the bottle *g* is slowly lowered until the surface of the water therein is on an exact level with 0 on the tube *ib*, the stop-cock *b* opened and the bottle *f* gradually raised until sufficient gas from *ah* has been transferred to *bi*, indicated by the volume taken reading from the mark 0 on the graduated tube *ib* to the mark 100 cc. immediately in contact with the stop-cock *b*.

Having thus obtained 100 cc. of the gas, the stop-cock *b* is closed and *f* is raised until all the remaining gas in *ah* and *ab* is displaced by the water. The first constituent of the gas to be determined is the carbon dioxide (CO_2). The gas is now transferred to the tube *ah* by raising *g* and opening *b*, keeping *a* closed and *f* lowered. When the water reaches *b* the latter is closed.

Fifty cc. of a solution of caustic potash are placed in the funnel cap *c*. (The solution being made by dissolving 280 grams of potassium hydrate in 1000 cc. of distilled water.)

Open the stop-cock *a* only partially, so that the solution of caustic potash in *c* may slowly drop down through the gas in the tube *ah* and absorb the carbon dioxide in so doing.

---

[1] The water used in this apparatus should contain 100 grams sodium chloride in each liter of distilled water.

When all the caustic potash in *c* (with the exception of two or three cc.) has passed through *a*, the latter is closed, thus preventing entrance of any air; *b* is opened, *f* is slowly raised and *g* lowered. Continue the raising of *f* until the water in the tube *ha* reaches the stop-cock *b* and immediately close the latter. Allow the gas to stand in the tube *ib* five minutes before taking the reading of the volume on the tube, bearing in mind that the level of the water in *g* must be on a level with the water in *ib* to obtain equal pressure. The difference between 0 and the point indicated by the water in the tube *ib* will give the amount of carbon dioxide absorbed from the gas by the caustic potash. Thus:

Original volume indicated at	0.0
After removal of carbon dioxide..........................	11.2

or 11.2 per cent. carbon dioxide *by volume*.

To obtain the oxygen the gas is forced from *ib* into *ah*, as before, and in *c* is placed fifty cc. of an alkaline solution of pyrogallic acid.

This latter solution is formed by dissolving ten grams of pyrogallic acid in twenty-five cc. of distilled water, placing it in *c* and adding thirty-five cc. of the caustic potash solution. This is allowed to pass slowly through *a* and gradually absorbs the oxygen in the gas. *a* is closed before all the liquid passes out of *c*. Repeat with the same quantity of alkaline pyrogallic solution. Transfer the gas in the usual manner to *ib*, and after allowing to stand five minutes, take the measurement thus:

Previous reading..................................	11.2 cc.
After absorbing oxygen..........................	19.6 "
Oxygen..................................	8.4 "

or 8.4 per cent. by volume.

Before transferring the gas to *ah* for the determination of the carbon monoxide, all the water in *f* and *ah* must be replaced by distilled water;[1] to do this, open the three way cock *e*, open *a*, and all the water can be caught in a large beaker at *e*. Wash

---

[1] Carbon dioxide is much more soluble in distilled water than carbon monoxide or nitrogen. For this reason the water used in the apparatus at the commencement of the gas analysis contains sodium chloride. After the determination of carbon dioxide distilled water can be used.

out *f* and *ah* three times with the water, then close *e* in the proper manner so that the water placed in *f* will rise in the tube *ha* to *a*, then close *a*, lower *f*, raise *g*, open *b*, placing the gas in *ah* for treatment with a solution of cuprous chloride to determine the carbon monoxide.

The cuprous chloride solution is made by dissolving thirty grams of cuprous oxide in 200 cc. hydrochloric acid (sp. gr. 1.19), and using fifty cc. as soon as the solution has reached the temperature of the room.

Experience has shown that a freshly made solution acts much better as an absorbent of carbon monoxide than one that has stood several days. Fifty cc. of this solution are placed in *c* and allowed to slowly drop through *a* and absorb the carbon monoxide as it passes through the gas. This absorption should be repeated at least three times. The heat generated during this absorption often causes such an increase in the volume of the gas that when the latter is transferred to the tube *ib* for measurement, the reading may prove minus. To insure accuracy proceed as follows:

The gas, after fifteen minutes, is transferred in the usual way to *bi*, and the water in *f* and *ah* is replaced with distilled water. The gas is now returned to *ah* and a solution of potassium hydroxide is placed in *c* and allowed to pass through the gas in *ah*, absorbing all traces of hydrochloric acid gas. Repeat with this once. Return the gas to *bi*, allow to stand fifteen minutes, then take the reading:

Previous reading	19.6	cc.
After using Cu_2Cl_2 solution	20.7	"
CO	1.1	"

The nitrogen is determined by subtracting the total amounts of carbon dioxide, oxygen and carbon monoxide from 100.

Thus the analysis will read:

Carbon dioxide	11.2	per cent. by volume.
Oxygen	8.4	" " " "
Carbon monoxide	1.1	" " " "
Nitrogen	79.3	" " " "
Total	100.0	" " " "

In this analysis no corrections are required for the tension of the aqueous vapor, since the original gas is saturated with moisture, and during the analysis all measurements are made over water.[1]

To convert percentages by volume to percentages by weight proceed as follows :

1 liter of oxygen	gas weighs	1.430	grams.
1 " " hydrogen	" "	0.0895	"
1 " " nitrogen	" "	1.255	"
1 " " air	" "	1.293	"
1 " " carbon dioxide	" "	1.996	"
1 " " carbon monoxide	" "	1.251	"
1 " " methane	" "	0.7151	"
1 " " acetylene	" "	1.252	"

Then	11.2 cc. carbon dioxide	gas weighs	0.02202	gram.
	8.4 " oxygen	" "	0 01201	"
	1.1 " carbon monoxide	" "	0.00138	"
	79.3 " nitrogen	" "	0.09952	"
	100.0 " ——	" "	0.13491	"

If the 100 cc. of gas weighs 0.13467 gram, then

$$\text{The carbon dioxide} = \frac{0.02202 \times 100}{0.13491} = 16.32 \text{ per cent. by weight.}$$

$$\text{The oxygen} = \frac{0.01200 \times 100}{0.13491} = 8.97 \text{ per cent. by weight.}$$

$$\text{The carbon monoxide} = \frac{0.00138 \times 100}{0.13491} = 1.02 \text{ per cent. by weight.}$$

$$\text{The nitrogen} = \frac{0.09952 \times 100}{0.13491} = 73.69 \text{ per cent. by weight.}$$

Total 100.00 per cent. by weight.

Analysis of Flue Gases with the Orsat-Müencke Apparatus.

Where the determinations to be made are the percentages of carbon dioxide, carbon monoxide, oxygen and nitrogen, this

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[1] The solubility of these four gases, at normal temperature and pressure, are as follows :

1 volume of air-free water at 15° C. absorbs	1.002	volume of carbon dioxide.
1 " " " " " " " "	0.024	" " carbon monoxide.
1 " " " " " " " "	0.030	" " oxygen.
1 " " " " " " " "	0.015	" " nitrogen.

apparatus offers many advantages over any other. It is shown in Fig. 69, and is thus described:

The measuring burette *A*, of 100 cc. capacity, is surrounded by a large cylinder filled with water, in order to free the gas from changes of temperature, and the first forty-five cc. are divided into tenths cc., the remaining fifty-five cc. into cubic centimeters. The thick capillary glass tube is fastened at both ends, at *i* in a cut of the dividing panel, and at *o* by means of a small brace, attached to the cover of the case.

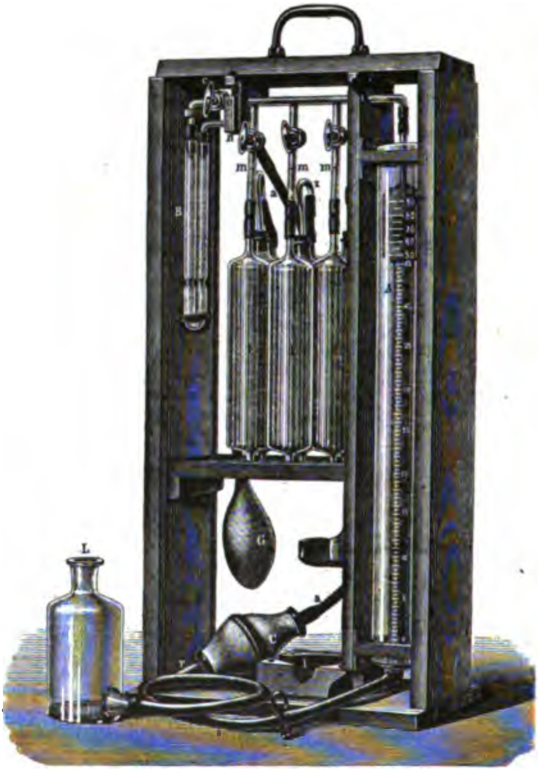

Fig. 69.

The capillary tube is bent at its further end and connected with the U tube *B*, containing cotton, and at the bend is filled with water in order to retain all dust and to saturate the gas thoroughly with moisture before measuring takes place.

The rear end of the three way cock *c* is connected by means of a rubber tube *a* with the rubber aspirator *C*, which fills the tube with the gas to be analyzed.

The absorption takes place in the "U" formed vessels *D*, *E*, and *F*, which are connected with the stoppers by short rubber tubes. For the enlargement of the absorbing surface, *D*, *E* and *F* are filled with glass tubes. Since the mark *m* is above the place of connection, the latter is always moistened by the respective liquid and therefore can easily be maintained air tight. The other end of the U tube vessel is closed by a rubber cork, which contains the small tube *x*; the small tubes are all connected to one rubber bulb of about 200 cc. capacity in order to keep out the atmospheric oxygen. The entire apparatus is enclosed in a wooden case fifty centimeters high and twenty-five centimeters wide. Its use is indicated as follows: The glass cylinder surrounding the burette *A* as well as the bottle *L* are filled with distilled water. In order to fill the three absorbing cylinders, the stoppers are removed as well as glass tubes *x* and the rubber bag *G*, and 110 cc. potassium hydroxide solution (sp. gr. 1.26) poured into the vessel *D*, so that the latter is about half full. This is for the absorption of the carbon dioxide. *E* contains a solution of eighteen grams of pyrogallic acid in forty cc. of hot water, which is poured into *E*, and then seventy cc. of potassium hydroxide solution (sp. gr. 1.26) added, whereby the oxygen is absorbed in the gas under examination.

The carbon monoxide is absorbed in the cylinder *F*, which contains a solution of cuprous chloride made as follows: Thirty-five grams of cuprous chloride are dissolved in 200 cc. hydrochloric acid (concentrated), fifty grams of copper clippings added and the mixture allowed to stand in a glass-stoppered bottle for twenty-four hours. Each glass tube in *F* contains a spiral of copper wire. 100 cc. of water is added to the solution (no precipitate forming), and enough is transferred to *F* to fill to the required point. The solutions in the rear section of *D*, *E*, and *F* are transferred to the front sections, where the absorption of the gas takes place as follows: The three glass stoppers are closed, the stop-cock *c* turned horizontal and the bottle *L*, containing distilled water, raised so that the water fills the burette *A*, give a quarter turn to the left to the stop-cock *c*. so that the second passage leads to the tube *B*, open the stop-cock of the vessel *D*, lower the bottle *L* and carefully open the pinch-cock placed on the tube *s*, so that potassium hydroxide solution rises to the mark *m*, whereupon the stop-cock is closed. The fluids of the two other absorbing vessels are raised in the same way to the mark *m*. The three stoppers with the glass tubes *x* are then attached. About one cc. of water is placed in the tube *B*, loose cotton placed in both sides, the stopper reinserted and connected with the tube *n*. After filling the burette *A* with water to the 100 cc. mark by raising the bottle *L*, the stop-cock is turned so that the connection of the rubber aspirator *C* with the chimney, containing the flue gases, is brought about through the tube *B*. Aspiration of the gas into the apparatus is now performed by compressing *C* ten or fifteen times till the whole conductor is filled with gas. This is easily done by compressing *C* with the left hand, closing the attached tube *r* with the thumb of the right hand, and then upon opening the left hand allowing *C* to expand, raising the thumb again, compressing *C*, etc., till the object is obtained. To fill the burette *A* with the gas, the stop-cock *c* is turned horizontal, the pinch-cock of the tube *s* opened, and the bottle *L* lowered until the gas reaches the zero point in *A*, whereupon *c* is closed.

To determine the carbon dioxide, the stop-cock of *D* is opened and *L* raised with the left hand, so that on opening the pinch-cock of *s* with the right, the gas enters the cylinder *D*; *L* is lowered again until the potassium hydroxide solution in *D* reaches to about the tube connection under *m*, and once again drives the gas into the potassium hydroxide vessel by the raising of *L*. This is repeated two or three times, and the gas returned to the burette *A* by opening the pinch-cock of *s* and raising *L*, and closing the glass stop-cock of *D*. To measure the amount of absorbed carbon dioxide, the bottle *L* is held next to the burette in such a way that the water stands at the same level in both vessels, the pinch-cock of *s* closed, and the remaining volume of gas read off. This amount subtracted from 100 cc. gives the amount of carbon dioxide. The gas is now passed into the vessel *E* in the same manner as in *D*, the oxygen being absorbed by the alkaline pyrogallate solution. This absorption must be repeated three or four times or until no diminution of volume takes place. The gas is then returned to the measuring burette *A* and the amount of absorption measured.

The gas is then passed into the vessel *F* for the absorption of carbon monoxide. After repeating for a number of times the absorption in *F* the gas is passed into *D* before measurement in *A* of the absorbed carbon monoxide. This is necessary on account of the vapors of hydrochloric acid retained by the gas after contact with the cuprous chloride solution in hydrochloric acid. After passing the gas into *D* three or four times, it is then measured as usual in *A*, the remaining gas being nitrogen.

The composition of the chimney gases is an index of the working of the furnaces under the boilers. When the fuel is properly consumed, the furnace gases should contain only nitrogen, oxygen, steam, and carbon dioxide, and to secure this result, excess of air is required, but this excess must not exceed a certain amount, otherwise too great a volume of air is heated and the heat wasted.

This excess of air can be determined by finding the amount of carbon dioxide in the furnace gases; thus the percentages of carbon dioxide, herewith given, show the amount of air used in the furnace.[1]

4 per cent. carbon dioxide indicates 4.9 times the theoretical amount of air required was in the gases.

5 per cent. carbon dioxide indicates 3.5 times the theoretical amount of air required was in the gases.

6 per cent. carbon dioxide indicates 3.0 times the theoretical amount of air required was in the gases.

7 per cent. carbon dioxide indicates 2.5 times the theoretical amount of air required was in the gases.

8 per cent. carbon dioxide indicates 2.3 times the theoretical amount of air required was in the gases.

9 per cent. carbon dioxide indicates 2.0 times the theoretical amount of air required was in the gases.

10 per cent. carbon dioxide indicates 1.7 times the theoretical amount of air required was in the gases.

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[1] Experiments at Munich: *Bayrisches Industrie und Gewerbeblatt*, 1880.

12 per cent. carbon dioxide indicates 1.5 times the theoretical amount of air required was in the gases.

17 per cent. carbon dioxide indicates 1.0 times the theoretical amount of air required was in the gases.

It is customary in boiler trials to make analyses of furnace gases and calculate the amount of air required for combustion from the percentage of carbon dioxide found in the furnace gases.

Prof. W. C. Unwin, F.R.S.,[1] states that this method is accurate in principle, but the samples analyzed are a very minute fraction of the total chimney discharge, and the samples may not be the average samples. What is wanted is an instrument as easily read as a pressure gauge, and giving continuous indications, such as the dasymeter of Messrs. Siegert & Durr, of Munich. (Fig. 70.) This is a fine balance in an enclosed case, through which a current of the furnace gases is drawn. At one end of the balance is a glass globe of large displacement, at the other a brass weight. Any change of density of the medium in the chamber disturbs the balance. A finger on the balance moving over a graduated scale gives the amount of the alteration of density.

Fig. 70.

An air injector draws the furnace gas from the flues, and it is

[1] *Nature*, (May 23, 1895), p. 89.

filtered before entering the balance case. An ingenious mercurial compensator counterbalances any effect due to change of temperature or barometric pressure.

The dasymeter is usually combined with a draught gauge, and an air thermometer or pyrometer in the flue is required if the amount of waste heat is to be calculated.

The losses through sensible heat in the escape gases can be easily determined with the assistance of the dasymeter and Siegert's approximate formula in the following way:

Let the carbon dioxide = x in per cent.
temperature of discharged gases = T (Celsius scale),
temperature of draught at grate = t,
then the loss of T with the coals as fuel equals:

$$V = 0.65\frac{T - t}{CO_2} \text{ in per cent. of the heat value.}$$

With lignite, peat, wood, etc., the coefficient varies according to the contents of water and the coefficient of heat of the fuel and is so much the greater, the less valuable the combustible is.

With coal furnaces the loss of heat can immediately be obtained from the following diagrams without any calculation:

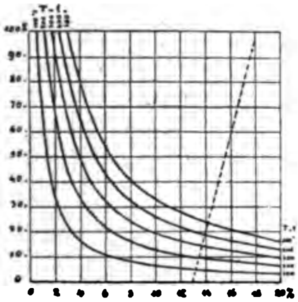

Fig. 71.

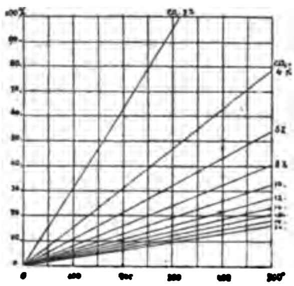

Fig. 72.

In Fig. 71 look up the carbon dioxide = contents of carbon dioxide in the lower horizontal (abscissa row), follow the vertical line appertaining thereto till it intersects the curve of the surplus temperature, draw from this point of section a horizontal line to the left and it will give the amount, per cent. of the loss of heat as indicated by that point on the scale at which it was intersected.

In Fig. 72 look up the amount, per cent. of the surplus temperature on the bottom abscissa line, raise a perpendicular line from the point till it intersects the line drawn diagonally for that amount of carbon dioxide indicated by the dasymeter. The horizontal line through this point of section indicates, on the scale for the loss of heat on the left, the loss to be determined. Points lying between two given abscissa can easily be assumed on both diagrams by eye measurement.

Experiments show that when using horizontal and step grate furnaces, as also the Ten-Brink furnaces, the most profitable combustion is obtained when about ten to fourteen per cent. of carbon dioxide is contained in the escaped gases, and in the use of gas furnaces about seventeen to eighteen per cent.

The dasymeter requires, initially, exceedingly delicate adjustment, and its indications must be checked from time to time by analysis of the gas. It is set to read zero with pure air, and then any increase of density due to carbon dioxide is read as a percentage on the scale. When in adjustment, it is as easy to read the percentage of carbon dioxide in the furnace gases as to read the pressure on a pressure gauge. When the dasymeter is fitted to a boiler, the stoker has directions to adjust the supply of air, so that the furnace gases have about twelve per cent. of carbon dioxide.

With practice he learns what alterations of the damper or fire-door, or thickness of fuel on the grate are necessary, or whether an alteration of grate area is desirable.

After a little practice the percentage of carbon dioxide can be kept very constant.

Uehling & Steinbach describe an instrument they make use of to determine the composition of furnace gases, and which indicates automatically and continually the percentages of carbon dioxide and monoxide present in furnace gases. It is fully described in United States patent No. 522746.

XXX.

Gas Analysis.

COAL GAS, WATER GAS, OIL GAS, PRODUCER GAS, ETC., BY MEANS OF THE HEMPEL APPARATUS.

In technical analysis of gases the most complete experiments may be conducted with the aid of the Hempel apparatus. The essential feature of this apparatus consists in the fact that measurements and absorptions may be conducted separately and in special apparatus. Gas burettes serve the first purpose and for the latter gas pipettes.[1] The gas burette, as shown in Fig. 73, consists of two parts, the calibrating tube *b* and the levelling tube *a*. The first has a constant diameter and ends above in a capillary tube about one-half millimeter in diameter and three centimeters in length; at the bottom it tapers into a small tube bent at an angle and passing through and protruding from the wooden stem *g*, supported by an iron base.

The calibrating tube is divided from the capillary part down to a little above the wooden support into two-tenths cc., the total graduation comprising 100 cc. A rubber tube about 120 cm. long, having a short length of glass tubing inserted at about the middle, as shown in Fig. 73, serves to connect the glass tube projecting at *g* with the levelling tube *a*, which at the bottom is similarly fastened into the base at *e*. The tube *a* at *h* widens into a funnel to facilitate pouring in the water. Over the capillary tube *c* of the measuring tube a short piece of heavy rubber tubing is fastened by means of wire. A strong "Mohr" pinch-cock *f* enables one to close the measuring tube directly above the capillary tube. The rubber tube at *d* has a ⊓ shaped capillary glass tube leading from it (see *E* Fig. 75), to provide for a connection with the various gas pipettes.

Since in this simple gas burette water is used as a sealing fluid, it is not adopted for the analysis of gases containing constituents easily soluble in water. In such cases Winkler's gas burette, Fig. 74, is used. The capillary tube *b* is closed below

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[1] *Chemisch-technische Analyse*, Post, pp. 117, *et. seq.*

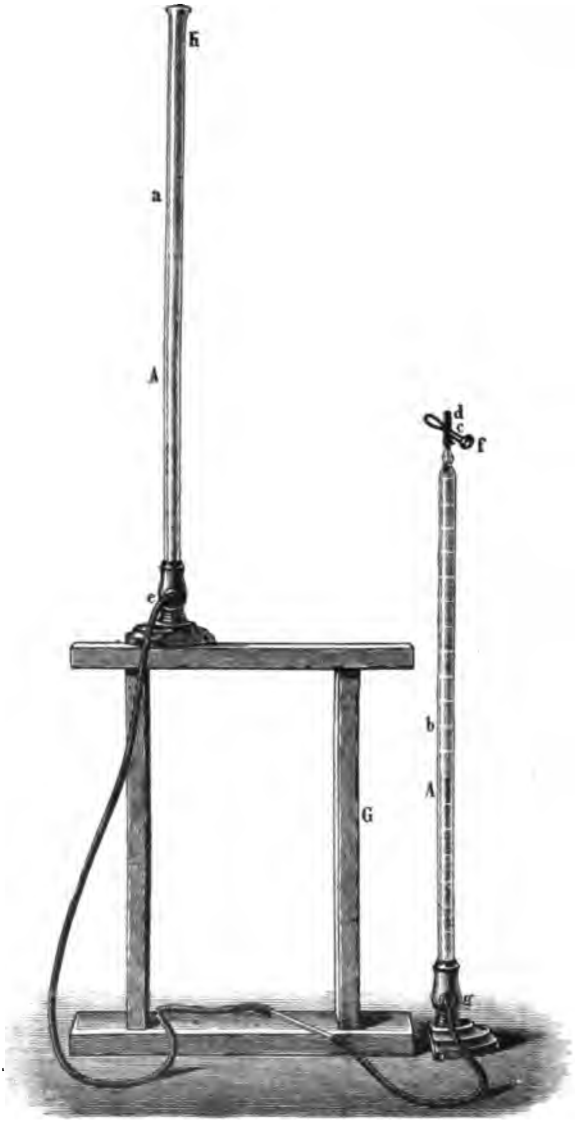

Fig. 73.

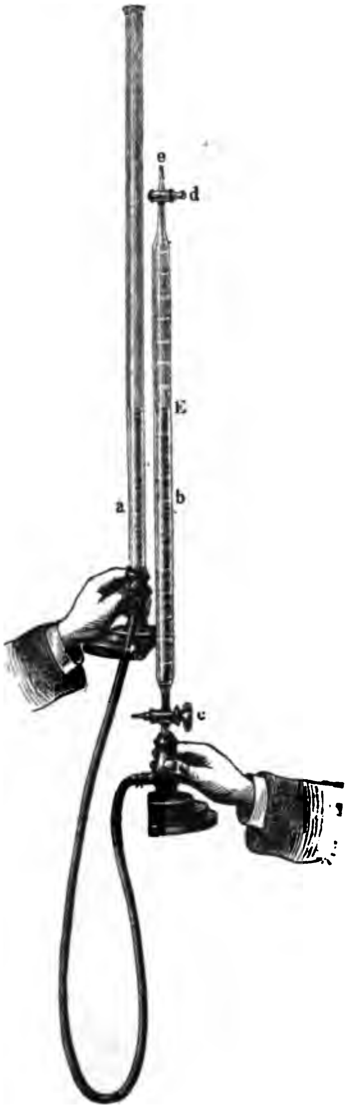

Fig. 74.

by a three-way cock *c* and above by means of a simple stop-cock *d*. Similarly to the Hempel burette both the measuring tube *b* and levelling tube *a* are fastened into iron stands and are connected by a rubber tube. The space between the stop-cocks *c* and *d* is divided into 100 cc., and each of these into fifths of cc. Before use the "Winkler" burette must be thoroughly dried, by rinsing with alcohol and ether, and thereupon passing a current of dry air through it. In order to admit a sample of gas to be analyzed, *e* is connected by rubber or glass tubing with the source of gas, and the length-bore of the three-way cock *c*, which communicates with the inside of *b*, is attached to an aspirator or rubber pump. Gas is drawn through till all the air has been displaced, thereupon closing *c* and *d*. In order to transfer the gas into the pipettes, the levelling tube *a* and the rubber tube are filled with water till the latter commences to flow from the stop-cock *c*, which at this moment communicates with *a* through its length-bore. The flow of water is checked by closing with a rubber tube and glass rod, or a pinch-cock. At Winkler's suggestion the calibrating tube *b* of Hempel's simple gas burette is surrounded by a water jacket in order to reduce the effect of atmospheric changes of temperature upon the gas in the burette.

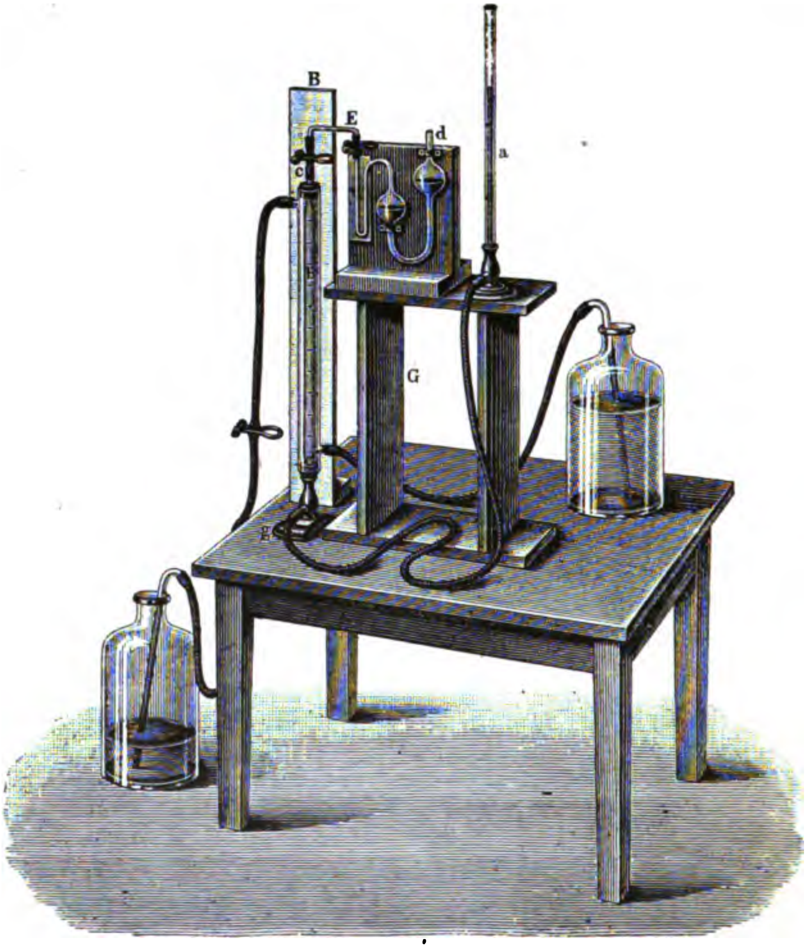

Fig. 75.

The larger glass tube serving as a water jacket, Fig 75, is closed above and below by two rubber corks, through which the calibrating tube passes, and has also above and below two small projecting glass tubes, used for filling or discharging the water; they are either simply closed by rubber corks or have attached to them rubber tubes to produce a continuous flow of water in the jacket.

On the working table there rests a stand *G* upon which the pipettes are placed and whose height is so adjusted that the entrance to the pipette and the capillary tube of the burette are at one level. These pipettes, which must be equal in number to the various absorptions which are to be executed (since each one remains charged with one liquid and always serves for the determination of only one gas constituent), have according to the purpose which they serve, different attachments.

The simple absorption pipette, Fig. 76, consists of two glass globes *a* and *b*, connected by means of a bent glass tube *d*, and fastened to a wooden stand to prevent breakage. A capillary tube *c* passes from the globe *b* before a plate of milk glass *m*, which is let into the wooden stand, in order to easily trace the movements of the liquid thread in the capillary tube *c*. The exit tube *f* of the globe *a* and the capillary tube *e* extend above the wooden frame; a small rubber tube *e* is connected to the protruding tube *c* and fastened by means of wire. The reagent to be used in the pipette is poured in at *f*, entirely filling the globe *b*, *a* only partially, and the capillary tube *c* to the junction with the rubber tube near *e*. When not in use, *f* is closed by a cork and *e* by a glass rod, which during use is displaced by a pinch-cock.

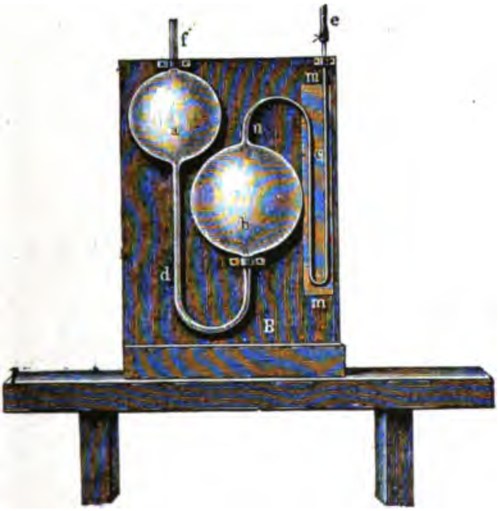

Fig. 76.

A label designating the contents of the pipette is attached to the wooden frame. The gas is transferred into these pipettes, brought into intimate contact with the reagent by shaking and thus freed from the constituent gas under consideration. The simple burette, containing caustic potash solution (1 to 2) is used for absorption of the carbon dioxide. The pipette containing fuming sulphuric acid, Fig. 77, is so modified that shaking is avoided. Above the globe *b*, filled with disulphuric acid, the smaller globe *g* also filled with the fuming sulphuric acid and pieces of broken glass (the latter placed there by the glass-blower). When the gas passes into the pipette it comes into contact with large surfaces of the broken glass, which are covered with the absorbing liquid. Passing the gas through *g* three or four times suffices for complete absorption. The heavy hydrocarbons in the gas are absorbed in the pipette by the disulphuric acid.

Fig. 77.

Fig. 78 shows the compound pipette, two of which are used:

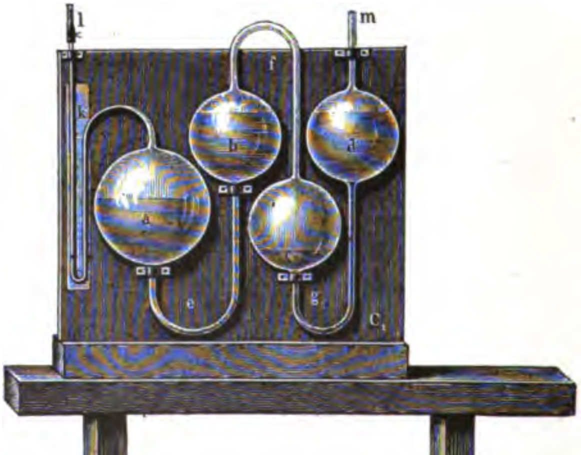

Fig. 78.

one for the determination of oxygen in the gas, the other for the determination of the carbon ~~diox~~ide. monoxide CO.

This pipette is charged with alkaline pyrogallate solution for the former and with cuprous chloride solution for the latter determination.

The bulb *a* next the capillary tube is filled with a solution of alkaline pyrogallol, the bulb *b* partially filled with the same solution, the bulb *c* is empty or nearly so, and the bulb *d* contains distilled water. The alkaline pyrogallol solution is made by dissolving one part of a twenty-five per cent. pyrogallol solution in water, in six parts of a sixty per cent. solution of caustic potash.

In order to illustrate the working of the Hempel apparatus, an analysis is here given of a gas containing carbon dioxide, oxygen, carbon monoxide, ethylene, methane, hydrogen, and nitrogen. A sample of this gas, 100 cc., is collected and measured in the gas burette. The carbon dioxide is first absorbed by passing the gas into the potassium hydroxide pipette, Fig. 76, containing a solution of one part potassium hydroxide[1] in two parts of water. Agitate the gas and potash solution, and after waiting five minutes pass the gas back into the measuring burette and determine the carbon dioxide absorbed. The contraction produced gives directly the percentage of carbon dioxide, since 100 cc. were used at starting.[2]

The oxygen is next absorbed in the compound pipette, Fig. 78. The absorption is complete in about five minutes. The amount of oxygen absorbed is now measured.[3]

Some chemists prefer to use stick phosphorus for the absorption of oxygen, The phosphorus pipette is shown in Fig. 79. The bulb b_1 contains pieces of phosphorus inserted through the opening at *k*, which is closed by a rubber stopper. The bulb b_1 and the capillary tube c_1 are filled with water, likewise a portion of a_1.

After the absorption of the oxygen the next step is to absorb the acetylene by means of disulphuric acid in the pipette, Fig.

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[1] Potassium hydroxide purified by alcohol cannot be used.

[2] Sulton's Vol. Anal., p. 522.

[3] Chromous chloride may also be used for the absorption of oxygen, even in the presence of hydrogen sulphide and carbon dioxide. *Liebig's Annalen*, 228, 112.

Fig. 79.

77. The absorption is complete in a few minutes, but the remaining gas, previous to measuring, should be passed into the potassium hydroxide pipette twice, in order to free the gas from fumes of sulphur trioxide. Allow the gas to stand in the graduated burette five minutes before taking measurement of volume. The

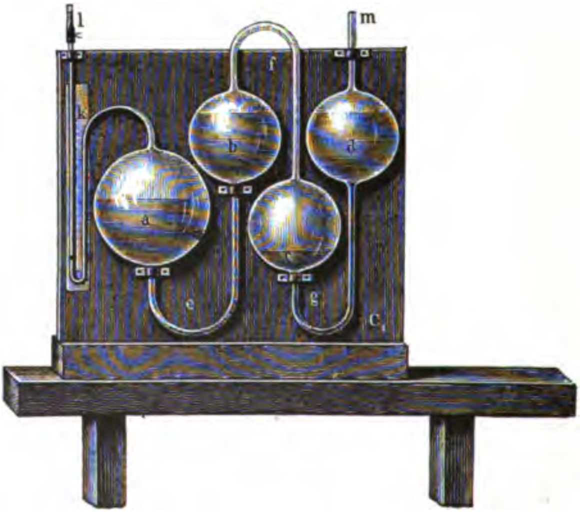

Fig. 80.

carbon monoxide is next absorbed by means of a solution of cuprous chloride (Cu_2Cl_2) in hydrochloric acid[1] in the compound pipette, Fig. 80.

Complete absorption of carbon monoxide is somewhat slow. Fifteen minutes should be given for this with frequent agitation of the gas with cuprous chloride solution. The gas is then passed into the potassium hydroxide pipette to absorb fumes of hydrochloric acid before it is transferred to the measuring burette, where after waiting five or ten minutes the volume can be measured. The residual gas now contains hydrogen, methane, and nitrogen.

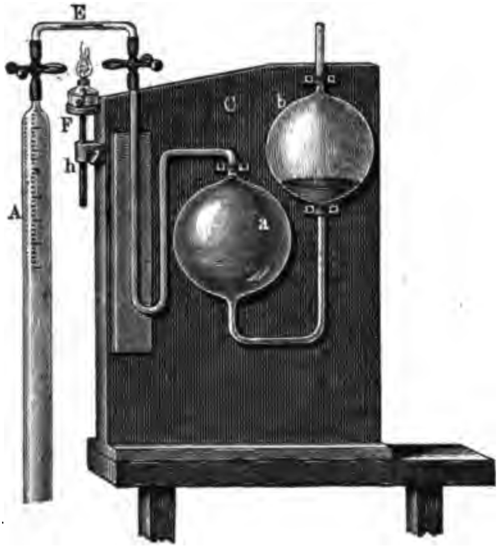

Fig. 81.

The hydrogen is determined by passing the gases over palladium sponge in the palladium tube *E*, Fig. 81.

An improved form is shown in Fig. 82.

Fig. 82.

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[1] Prepared by dissolving sixty grams of Cu_2O (red oxide of copper), in 400 cc. of hydrochloric acid, sp. gr. 1.19. One cc. of this fresh solution will absorb twenty cc. of carbon dioxide.

The palladium tube is kept at a temperature not exceeding 100° C, either by using a minute flame as shown in Fig. 81, or by immersing the tube, Fig. 82, in a beaker of water at the required temperature. Under these conditions the combustion of hydrogen proceeds without the combustion of any methane. The gas is passed and repassed through the tube *slowly* at least three times, the palladium tube during the whole operation being connected with an ordinary absorption pipette filled with water, Fig. 81.

Fig. 83.

Finally the gas is transferred to the measuring burette and the volume determined. As the hydrogen has burned to water by uniting with the occluded oxygen in the palladium sponge, the diminution in volume represents the amount of hydrogen in the gas directly. The residual gas now contains methane and nitrogen. Of this gas ten cc. are now taken and the rest allowed to escape, or if necessary can be collected and saved in a pipette.

To the ten cc. of the gas in the burette ninety cc. of air are added and this mixture of air, methane, and nitrogen passed into the explosion burette, Fig. 83.

The gases are thoroughly mixed and then exploded. The current required to do this is generated by a dip battery of four cells connected with a small induction coil, and with the explosion pipette by the platinum wires *kk*, Fig. 83, which are fused into the pipette. After the explosion the gases are led into the potassium hydroxide pipette to absorb the carbon dioxide formed by the combustion of the methane. One volume of methane requires four volumes of oxygen for its combustion, producing one volume of carbon dioxide and two volumes of water. One-third of the loss of volume, after the explosion and measurement in the burette, gives the volume of methane in the ten cc. of the gas. This amount subtracted from ten cc. gives the amount of nitrogen in the ten cc., and these amounts must be calculated back into values of the total amount of gas left in the burette before mixing with air.

The following analysis of a sample of carburetted water gas will indicate the working of the method.

100 cc. of the gas taken.

Before use of KOH	100.0 cc.
After “ “ “	96.2 “
CO_2 =	3.8 “.

No oxygen present.

Before use of $H_2S_2O_7$	96.2 cc.
After “ “ “	81.6 “
(Illuminants,) C_2H_4, etc. =	14.6 “
Before use of Cu_2Cl_2 solution	81.6 cc.
After “ “ “ “	53.6 “
CO =	28.0 “
Before use of Palladium tube	53.6 cc.
After “ “ “ “	18.0 “
H =	35.6 “

$CH_4 + N$ remaining = 18 cc.

Ten cc. taken + ninety cc. air. After explosion in the explosion pipette (Fig. 78) and measurement after absorption of carbon dioxide formed, the volume was 71.8 cc., which corresponds to 28.2 cc. of absorption or nine and three-tenths cc. of methane. If ten cc. of the gas gave nine and three-tenth cc. of methane, eighteen cc. of the gas (amounts remaining before mixing with air) will give 16.7 cc. of methane.

	Calculated to 18 cc.
The volume of gas before adding air	18.0 cc.
" " " " after explosion and absorption (N)	1.3 "
	$CH_4 = 16.7$ "

Resumé :

Carburetted water gas.	By volume.	
CO_2	3.8	per cent.
(Illuminants) C_2H_4, etc	14.6	" "
CO	28.0	" "
H	35.6	" "
CH_4	16.7	" "
N	1.3	" "
Total	100.0	" "

and by weight :[1]

CO_2	9.6	per cent.
(Illuminants) C_2H_4, etc	23.7	" "
CO	45.1	" "
H	4.1	" "
CH_4	15.4	" "
N	2.1	" "
Total	100.00	" "

Some chemists prefer to determine the hydrogen and methane by explosion, instead of using the palladium tube for the hydrogen. In this case suppose a partial analysis of gas gave as follows (100 cc. of gas taken) :

Carbonic acid	2.2	per cent.
Oxygen	0.0	" "
Illuminants	12.8	" "
Carbon dioxide	24.2	" "
	39.2	" "

The remaining constituents being (in the 60.8 cc. gas left) methane, hydrogen and nitrogen. These are treated as follows:

Fifteen cc. of this residual gas are taken and mixed with eighty-five cc. of air. This is then passed into the explosion

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[1] For method of calculation see Analysis of Chimney Gases, p. 237.

burette containing water previously saturated with the gas. It is well shaken to insure thorough mixture of the air and gas, and then exploded by means of a spark from the induction coil. After fifteen minutes the reading is taken: the latter being 77.4 cc. or 22.6 cc. contraction. $(100 - 77.4 = 22.6.)$

Methane produces an equal volume of carbon dioxide by combustion; therefore, if the carbon dioxide produced be measured by absorption with potassium hydroxide, the amount represents the methane. Pass the gas into the potassium hydroxide burette and determine carbon dioxide.

77.4 cc. — 73 cc. = 4.4 cc. methane in the fifteen cc. of the gas mixed with the air, or in per cent. of 100 cc. of original gas:

$$15 \text{ cc.} : 60.8 :: 4.4 : x \text{ or } 17.83 \text{ per cent. methane.}$$

The hydrogen is determined as follows:

Let $C =$ contraction (15 cc. gas + 85 cc. air) after explosion.
" $D = CO_2 = CH_4$ in 15 cc. of gas.

$$\text{Then } H = \frac{2C - 4D}{3} = \frac{45.2 - 17.6}{3} = \frac{27.6}{3} = 9.2 \text{ per cent.}^1$$

hydrogen in fifteen cc. of the residual gas, or 32.07 per cent. hydrogen in the 100 cc. of the original gas.

By adding together all of the constituents determined, and subtracting this amount from 100, the residue is nitrogen. Thus the complete analysis will be:

CO_2	2.20	per cent.
Illuminants	12.80	" "
O	0.00	" "
CO	24.20	" "
CH_4	17.83	" "
H	37.95	" "
N	5.02	" "
Total	100.00	" "

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[1] Consult *Gasometrische Methoden*, R. Bunsen, 2nd edition, pp. 48–51.

XXXI.

Heating Value of Combustible Gases.

In the calculation of the fuel value of gases, the method as given by H. L. Payne[1] will be found accurate and convenient. Since the results of gas analyses are stated volumetrically the calculation of a number of analyses is greatly facilitated if the calories per kilo are converted into heat units per volume, and custom requires results to be stated in "B. T. U." per cubic foot of gas. Calories per kilo, multiplied by $\frac{9}{5}$ (the ratio of the Fahrenheit thermometer degree to the Centigrade) will give British thermal units per pound. Dividing this result by the number of cubic feet of each gas per pound will give "B. T. U." per cubic foot. To calculate the cubic feet per pound of gas the following fundamental relations are used:

1 pound = 453.59 grams.
1 meter = 39.37 inches.

1728 (cubic inches to one cubic foot) divided by $(3.937)^3 = 28.317$; therefore one cubic foot = 28.317 liters. From this is obtained: "B. T. U." per cubic foot = Calories per kilo $\times \frac{9}{5} \times \frac{28.317}{453.59} \times$ the liter weight in grams of the gas in question.

The following table contains the liter weights as determined by actual weight of some of the gases:

Gas.	Grams per liter.
H	0.0895
O	1.430
N	1.257
Air	1.293
CO	1.251
CO_2	1.966
CH_4	0.7155
C_2H_4	1.252

For the other hydrocarbons and gases not given above, we may substitute in the formula in place of liter weight the ex-

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[1] *J. Anal. Chem.*, 7, 230–235.

pression $\left(\frac{\text{molecular weight}}{2} \times \frac{0.08950}{1.007}\right)$. This formula then may be reduced to the following form: "B. T. U." per cubic foot $=$ calories (per kilo) $\times \frac{\text{molecular weight}}{2} \times 0.01003$, which is comparatively simple and can be used in all cases without very serious error.

Applying this formula to the different combustible gases we obtain

TABLE OF HEATING VALUES OF GASES.

One kilo of H evolves upon complete combustion 34,500 calories, or 62100 B. T. U. per pound, or 348 B. T. U. per cubic foot at 0° C. and 760 mm. pressure.

One kilo of CO evolves upon complete combustion 2,487 calories, or 4,476 B. T. U. per pound, or 349 B. T. U. per cubic foot at 0° C. and 760 mm. pressure.

One kilo of CH_4[1] (methane) (marsh gas) evolves upon complete combustion 13,245 calories, or 23,851 B. T. U. per pound, or 1,065 B. T. U. per cubic foot at 0° C. and 760 mm. pressure.

One kilo of C_2H_2 (acetylene) evolves upon complete combustion 11,925 calories, or 21,465 B. T. U. per pound, or 1,555 B. T. U. per cubic foot at 0° C. and 760 mm. pressure.

One kilo of C_2H_4[2] (ethylene) (olefiant gas) evolves upon complete combustion 11,900 calories, or 21,440 B. T. U. per pound, or 1,673 B. T. U. per cubic foot at 0° C. and 760 mm. pressure.

One kilo of C_2H_6 (ethane) (ethyl hydride) evolves upon complete complete combustion 12,350 calories, or 22,230 B. T. U. per pound, or 1,858 B. T. U. per cubic foot at 0° C. and 760 mm. pressure.

One kilo of C_3H_8 (propane) (propyl hydride) evolves upon complete combustion 12,028 calories, or 21,650 B. T. U. per pound, or 2,654 B. T. U. per cubic foot at 0° C. and 760 mm. pressure.

One kilo of C_3H_6 (propylene) evolves upon complete combustion 11,900 calories, or 21,420 B. T. U. per pound, or 2,509 B. T. U. per cubic foot at 0° C. and 760 mm. pressure.

One kilo of C_4H_{10} (quartane) (butane) evolves upon complete combustion 11,850 calories, or 21,330 B. T. U. per pound, or 3,447 B. T. U. per cubic foot at 0° C. and 760 mm. pressure.

One kilo of C_5H_{12} (quintane) (pentane) evolves upon complete combustion 11,770 calories, or 21,186 B. T. U. per pound, or 4,250 B. T. U. per cubic foot at 0° C. and 760 mm. pressure.

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[1] The heat values in calories of CH_4......C_3H_6 are taken from Thomson's *Thermochemie Untersuchungen*.

[2] The "illuminants" in water gas are (often) taken as a mixture of C_2H_4 and C_3H_6, and the B. T. U. per cubic foot as 2,000, which is about the mean of the two gases.

One kilo of C_6H_{14} (sextane) evolves upon complete combustion 11,620 calories, or 20,916 B. T. U. per pound, or 5,012 B. T. U. per cubic foot at 0° C. and 760 mm. pressure.

One kilo of C_6H_6 (benzene) evolves upon complete combustion 10,250 calories, or 18,450 B. T. U. per pound, or 4,010 B. T. U. per cubic foot at 0° C. and 760 mm. pressure.

One kilo of $C_{10}H_8$ (naphthalene) evolves upon complete combustion 9,620 calories, or 17,316 B. T. U. per pound, or 6,176 B. T. U. per cubic foot at 0° C. and 760 mm. pressure.

To calculate the heat units of a gas from its analysis, multiply the per cent. of each constituent by its number as given in the above table, and the sum of the products will represent the British thermal units evolved by the combustion of one cubic foot of the gas. Ordinary gas analysis includes as combustibles only hydrogen, carbon monoxide, methane, and "illuminants," the latter term representing the hydrocarbons that are determined by absorption in fuming sulphuric acid or bromine. This has proven to be a very trustworthy value where the hydrocarbons are derived chiefly from the decomposition of mineral oil, but if produced by the distillation of coal, this value is too low, owing to a larger percentage of benzene vapors contained.

The experimental conditions necessary to give these theoretical results are that the gas be measured at 32° F. and 760 B. and is burned with exactly the proper quantity of oxygen, and that the products of combustion are reduced to the initial temperature, the water being all in the liquid state. It is superfluous to say that this cannot actually be done; but as the whole matter is a theoretical discussion, it is decided to adhere to the scientific standard, and to state results in accordance with its definitions.

But in order to obtain figures which shall more nearly agree with practice, many persons have preferred to make their calculations under certain assumed conditions. This plan is not without merit, since by it a somewhat better idea of the true relative values of fuel constituents is obtained, similar conditions affecting different gases unequally. The following examples illustrate this:

The temperature for standard gas measurement in this country is 60° F., and this point is usually assumed as the initial temperature. As a final temperature in this case let the temperature of the steam be 100 pounds absolute pressure per square inch (328° F.), a point considerably lower than the average chimney flue heat. Under these conditions combustion taking place in air, not in oxygen, we must add the heat brought in by the gas and air at 60° F. and subtract the heat carried away by the products of combustion at 328° F., and since the volume of gas is greater at 60° than at 32°, we correct for this by multiplying the result $\frac{492}{492 + 28}$ or $\frac{492}{520}$.

The composition of air is:

	By volume.	By weight.
O	20.92 per cent.	23.134 per cent.
N	79.08 " "	76.866 " "

Hence 4.78 volumes of air contain one volume of oxygen, or one volume of oxygen is accompanied by 3.78 volumes of nitrogen.

The specific heats of the several gases are as follows:

Gas.	Sp. heat.
H	3.4
O	0.22
N	0.24
Air	0.24
CO	0.25
CO_2	0.22
CH_4	0.60
"Illuminants"	0.41

It will be more convenient in these computations to make use of the so-called "volumetric" specific heats, *i. e.*, the heat necessary to raise the temperature of one cubic foot of gas from 32° F. to 33° F.

Gas.	Vol. sp. heat.
H	0.019
N	0.019
O.	0.019
Air	0.019
CO	0.019
CO_2	0.027
CH_4	0.027
"Illuminants"	0.040

Then:

$2H + O + 3.78\ N = H_2O + 3.78\ N$; or 1 cu. ft. H + 2.39 cu. ft. air = 1 cu. ft. steam + 1.89 cu. ft. N.

The heat gained or brought in is as follows:

$H = 1 \times 0.019 \times [28 = (60^\circ - 32^\circ)] = 0.53$ B. T. U.
Air = 2.39 × 0.019 × 28 = 1.27 " " "

Total gain 1.80 " " "

The heat lost or carried away by the products of combustion is as follows: Water at 32° F. converted into steam at 328° F. absorbs 1,182 B. T. U. per pound, and one cubic foot of hydrogen produces when burnt 0.0502 pounds of water.

Steam = 0.0502 × 1182 = 59.4 B. T. U.
N = 1.89 × 0.019 × 296 = 10.6 " " "

Total loss = 70.0 " " "
Subtract gain = 1.8 " " "

Net loss = 68.2 " " "

348 B. T. U. less 68.2 B. T. U. leaves 279.8 B. T. U. and corrected for volume gives 264 B. T. U., a loss of twenty-four per cent.

In the case of carbon monoxide no water is produced by combustion and the former value is consequently much less affected.

One cubic foot of carbon monoxide + 2.39[1] cubic foot of air = one cubic foot carbon dioxide + 1.89 cubic foot nitrogen. Heat gained or brought in by carbon monoxide and air, the same as hydrogen and air in the previous case, one and eight-tenths B. T. U.

Heat lost:

$CO_2 = 1 \times 0.027 \times 296 =$ 8.0 B. T. U.
N as above = 10.6 " " "

Total loss 18.6 " " "
Subtract gain 1.8 " " "

Net loss 16.8 " " "

349.5 B. T. U. less 16.8 B. T. U. leaves 332.7 B. T. U.

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[1] Refer to sample in hydrogen combustion.

This corrected for volume gives 315, a loss of only ten per cent.

For marsh gas, 1 cubic foot $CH_4 + 4(2.39$ cubic foot air$) =$ one cubic foot $CO_2 + 2$ cubic feet steam $+ 4(1.89$ cubic feet $N)$.

Heat gained:

$CH_4 = 1 \times 0.027 \times 28\ (60^\circ - 32^\circ) =$ 0.8 B. T. U.
Air $= 4 \times 1.27$ B. T. $=$ 5.1 " " "

Total gain $=$ 5.9 " " "

Heat lost:

$CO_2 = 1 \times 0.027 \times 296\ (328^\circ - 32^\circ) =$ 8.0 B. T. U.
Steam $= 2 \times 59.4$ B. T. U. $=$ 118.8 " " "
$N = 4 \times 10.6$ B. T. U. $=$ 42.4 " " "

Total loss $=$ 169.2 " " "
Subtract gain $=$ 5.9 " " "

Net loss $=$ 163.3 " " "

1065 B. T. U. less 163.3 B. T. U. leaves 901.7 B. T. U.

This corrected for volume gives 853 B. T. U. $\left(901.7 \times \frac{492}{520}\right)$ a loss of twenty per cent.

For "illuminants" fifteen per cent. is taken as a fair loss, and the values are:

Gas.	32° F. initial. 32° F. final,	60° initial. 328° final.	Loss in per cent.
H..............	348.0 B. T. U.	264 B. T. U.	24
CO..............	349.5 " " "	315 " " "	10
CH_4.............	1065.0 " " "	853 " " "	20
Illuminants.....	2000.0 " " "	1700 " " "	15

Natural gas has been taken as a standard for heating gases with a valuation of 1000 B. T. U. per cubic foot.

At ninety-four per cent. CH_4, which is not far from the average, it will show by calculation 1000 B. T. U. per cubic foot and hence the numerical result obtained by this method for any fuel gas will indicate also its standing in that scale.

Referring to the analyses of gas given on page 256, the "B. T. U." per cubic foot are as follows:

PRODUCTS OF COMBUSTION CONDENSED.

	CO	28.0 per cent.	× 349.5	= 97.86 B. T. U.
	CO_2	3.8		
(Illuminants)	C_2H_4, &c	14.6 " "	× 2000.0	= 292.00 " " "
	H	35.6 " "	× 348.0	= 123.88 " " "
	CH_4	16.7 " "	× 1065.0	= 177.85 " " "
			Total	= 691.59 " " "

The " B. T. U." per cubic foot of gas will be as follows:

PRODUCTS OF COMBUSTION IN VAPOR AT 328° F.

	CO	28.0 per cent.	× 315	= 68.20 B. T. U.
	CO_2	....		....
(Illuminants)	C_2H_4	14.6 " "	× 1700	= 248.20 " " "
	H	35.6 " "	× 264	= 63.98 " " "
	CH_4	16.7 " "	× 853	= 142.45 " " "
	N	....		....
			Total	= 552.83 " " "

In determining the B. T. U. per pound or of calories per kilo, the analysis of the gas by weight is taken and not by volume, as just instanced.

The B. T. U. per pound of the gas would be :

PRODUCTS OF COMBUSTION CONDENSED.

	CO	45.1 per cent.	× 4476	= 2018.6 B. T. U.
(Illuminants)	C_2H_4, &c	23.7 " "	× 21440	= 5081.2 " " "
	CO_2			
	H	4.1 " "	× 62100	= 2546.1 " " "
	CH_4	15.4 " "	× 23851	= 3673.0 " " "
	N			......
			Total	= 13318.9 " " "

and where the products of combustion are in vapor at 328° F., as follows :

	CO	45.1 per cent.	× 3402.0	= 1534.3 B. T. U. per pound.
(Illuminants)	C_2H_4	23.7 " "	× 18224.0	= 4319.0 " " " " "
	CO_2			
	H	4.1 " "	× 47196.0	= 1935.0 " " " " "
	CH_4	15.4 " "	× 19081.0	= 2938.4 " " " " "
	N			
			Total	= 10726.7 " " " " "

MANUFACTURE OF WATER GAS.

Nearly all of the carburetted water gas in the United States is made either by the Lowe process or the Wilkinson process, probably four-fifths by the former.

Briefly stated, the Lowe water gas system consists in the decomposition of steam at a high temperature by incandescent carbon, thereby producing hydrogen and carbon dioxide: $2H_2O + C = 2H_2 + CO_2$.

In an excess of carbon, the carbon dioxide saturates itself with another carbon atom, forming carbon monoxide ($CO_2 + C = 2CO$, making the finished product $2H_2 + 2CO$).

In practical working the reduction of carbon dioxide to monoxide is never quite perfect, the unpurified gas usually containing about three per cent of carbon dioxide, to be extracted (as in coal gas) by lime purification.

As the gas, in the process of manufacture, passes from the generator to the carburetters, it is enriched by means of crude oil or cheaper distillates: hence the name *carburetted* water gas.

The generator, carburetter, and super-heater are cylindrical steel shells, thickly lined with special fire blocks, between which and the metal are annular spaces packed with non-conducting material. The generator is usually supported on short columns, as illustrated, leaving cartage room under the hopper-shaped ash-pit. The grate, controlled by the several cleaning doors, is located slightly above the ash-pit, and the fire is charged with coke through the door in the extreme top.

The generator is connected, both above and below the fuel-bed, with the top of the carburetter, the bottom of which leads laterally into the adjoining super-heater. The carburetter and super-heater, often referred to as the fixing-chambers,'' are filled with checker work, and affording such an enormous heating surface that even the heaviest distillates can be permanently gasified at the low temperatures necessary to the highest illuminating effect. The enriching oil is introduced at the top of the carburetter.

The oil heater is a simple and practical arrangement for pre-

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[1] Humphreys & Glasgow: "*Carburetted Water Gas*," 1895.

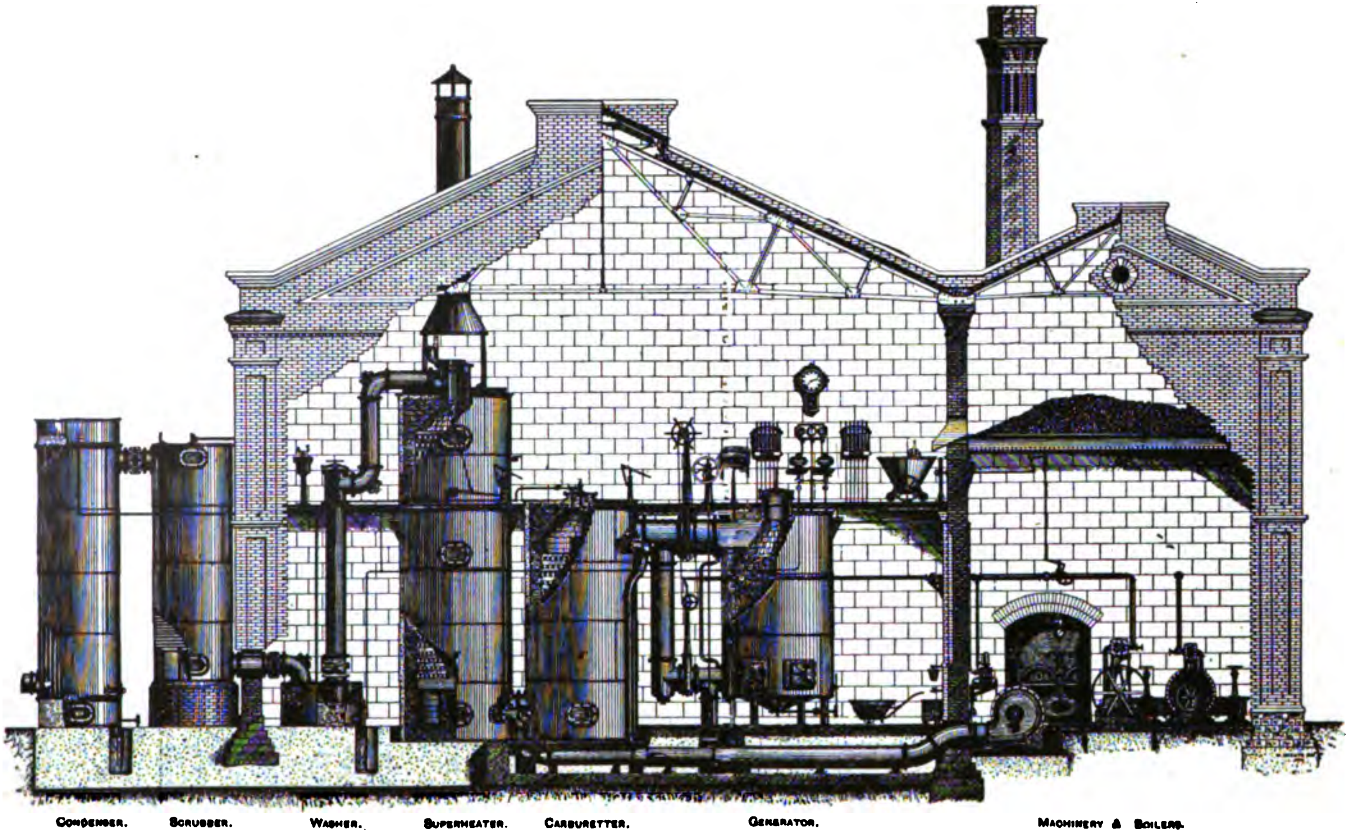
CONDENSER.
SCRUBBER.
WASHER.
SUPERHEATER.
CARBURETTER.
GENERATOR.
MACHINERY & BOILERS.

Fig. 84.

heating the oil on its way to the carburetter by means of the hot gas escaping from the super-heater.

Operation.—A fire is started in the generator, which is then deeply charged with coke and opened to the blast. The air enters in large volume below the grate and quickly kindles the fuel, while the hot products resulting from the partial combustion pass forward through the carburetter and super-heater and, after parting with their sensible heat, escape into the stack. As soon as these generator gases have sufficiently warmed the checker-work, supplies of secondary air are admitted to the top of the carburetter and the bottom of the super-heater respectively, and the combustion regulated to give the requisite temperatures in the two vessels simultaneously. The generator fire being in proper condition, and the carburetter and super-heater at the desired temperatures, the apparatus is ready for gas making. The blasts are shut off one by one, beginning with that of the super-heater; the stack valve is closed; steam is admitted under the fuel bed, and having traversed it, passes as water gas into the top of the carburetter. At this point the oil is introduced, and encountering the heated checker-work is vaporized and ultimately gasified in presence of the hot water gas. This process continues until the temperatures of the fire and the checker-work are sufficiently reduced. The oil is then shut off; next the steam; and the stack valve being opened the blasts are again admitted and the energy of the fire and the checker-work recuperated as first described. The generator is supplied with fuel at intervals of from forty-five to sixty minutes, and cleaned usually once during each shift. The gas passes from the seal through the scrubbers and condensers and is subsequently deprived of its carbon dioxide and treated for its slight sulphur impurities in the manner common to coal gas.

Uncarburetted water gas has the following composition :[1]

H	49.32	per cent.
CH_4	7.65	" "
CO	37.97	" "
CO_2	0.14	" "
N	4.79	" "
O	0.13	" "
Total	100.00	" "

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[1] King's Treatise on Coal Gas, 3, 362.

and after carburetting

H	38.05	per cent.
CH_4	11.85	" "
CO	29.40	" "
O	0.10	" "
CO_2	0.10	" "
N	3.71	" "
Illuminants	16.79	" "
Total	100.00	" "

The heating power of the uncarburetted gas per cubic foot would be:

					Products condensed.	
H	0.4932	×	348.0	=	171.63	B. T. U.
CH_4	0.0765	×	1065.0	=	81.47	" " "
CO	0.3797	×	349.56	=	132.72	" " "
Total					385.82	" " "

and the heating power of the carburetted water gas per cubic foot would be:

					Products condensed.	
H	0.3805	×	348.0	=	131.31	B. T. U.
CH_4	0.1185	×	1065.0	=	126.20	" " "
CO	0.2940	×	349.56	=	102.77	" " "
CO_2	....		....		....	
O	....		....		....	
N	....		....		....	
Illuminants	0.1679	×	2000.0	=	335.80	" " "
Total					696.08	" " "

An analysis of a sample of London (Eng.) coal gas gives the following:

H	27.70	per cent.
CH_4	50.00	" "
CO	6.80	" "
C_2H_4	13.00	" "
N	0.40	" "
O	....	" "
CO_2	0.10	" "
Aqueous vapor	2.00	" "
Total	100.00	" "

The heating power will be, per cubic foot,

Products condensed.
H .... 0.2770 × 348.0 = 96.39 B. T. U.
CH_4 .... 0.5000 × 1065.0 = 532.50 " " "
CO .... 0.0680 × 349.56 = 23.77 " " "
C_2H_4 .... 0.1300 × 1673.0 = 217.49 " " "

Total .... 870.15 " " "

and when the products of combustion are in a state of vapor (for instance 328° F.) the heating power per cubic foot will be:

H .... 0.2770 × 264 = 73.12 B. T. U.
CH_4 .... 0.5000 × 853 = 426.50 " " "
CO .... 0.0680 × 315 = 21.42 " " "
C_2H_4 .... 0.1300 × 1400 = 182.00 " " "

Total .... 703.04 " " "

There are few *complete* analyses of purified coal gas known,[1] *i. e.*, Heidelberg gas by R. Bunsen, Königsberg gas by Blochmann, and Hannover gas by Dr. Fischer.

	Heidelberg gas.	Königsberg gas.	Hannover gas. I.	Hannover gas. II.
C_6H_6	1.33	0.66	0.69	0.59
C_3H_6	1.21	0.72	0.37	0.64
C_2H_4	2.55	2.01	2.11	2.48
CH_4	34.02	35.28	37.55	38.75
H	46.20	52.75	46.27	47.60
CO	8.88	4.00	11.19	7.42
CO_2	3.01	1.40	0.81	0.48
O	0.65	....	trace	0.02
N	2.15	3.18	1.01	2.02
Total	100.00	100.00	100.00	100.00

In the Wilkinson process the water gas is made by the combined generator and retort process. (A full description of a recent plant will be found in the *The American Gas Light Journal*, **57**, 399, 401.

An analysis of a sample of Wilkinson water gas, made by the writer,[2] gave as follows:

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[1] Wagner's *Manual of Chemical Technology*, (13th edition, 1892) p. 39.

[2] Wood's " Thermodynamics, Heat Motors, and Refrigerating Machines, 3rd edition, pp. 260–261.

H	39.50	per cent.
Heavy hydrocarbons, Illuminants, C_3H_6 average	6.60	" "
CH_4	37.30	" "
CO	4.30	" "
N	8.20	" "
O	1.40	" "
Impurities (H_2O, CO_2 H_2S)	2.70	" "
	100.00	

One cubic foot containing 755.31 B. T. U. products condensed.

G. Lunge[1] gives an analysis of "Tessie du Motay" gas, as follows:

CO_2	——	
Illuminants	14.3	per cent.
O	0.6	" "
CO	27.7	" "
H	28.8	" "
CH_4	25.5	" "
N	3.1	" "
Total	100.0	" "

containing 827.62 B. T. U. per cubic foot, products condensed.

For complete details regarding the manufacture of coal gas consult King's *Treatise on Coal Gas*," edited by Thomas Newbigging, London.

PRODUCER GAS.

Constituents.	Sieman's gas.	Anthracite producer gas.	Soft coal producer gas.
CO	23.7	27.0	27.0
H	8.0	12.0	12.0
CH_4	2.2	1.2	2.5
CO_2	4.1	2.5	2.0
N	62.0	57.3	56.5
	100.00	100.00	100.00

The heating power of the Sieman's producer gas will be 134.1 B. T. U. per cubic foot: of the anthracite producer gas 153.7 B. T. U. per cubic foot, and of the soft coal producer gas 168.1 B. T. U. per cubic foot (products of combustion condensed).

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[1] "Wassergasfabrikation in New York," *Zeitschrift für angewandte Chemie*, 1894, pp. 137–142.

OIL GAS.

Oil gas is usually formed by vaporization of mineral oil at high temperatures. Two processes are in use: the "Pintsch" and the "Keith," the former probably representing ninety per cent. of the production of oil gas.

For a description of the "Pintsch" oil gas apparatus consult *Wagner's Chemical Technology* (edition of 1892), p. 80, also *J. Soc. Chem. Industry*, **6**, March, 1887. In the manufacture of Pintsch oil gas, in the United States, "mineral seal" oil is often used. This oil is a petroleum product having a specific gravity of about 0.840, flashing point 265° F., and fire test 311° F.

Several analyses, by the author,[1] of this oil give carbon 83.30 per cent., hydrogen 13.20 per cent., the remainder being oxygen, nitrogen, etc., and the analysis of the gas therefrom gave:

CO	0.5 per cent.
CH_4	57.7 " "
H	3.4 " "
(Illuminants) Benzene vapor, C_6H_6 Propylene C_3H_6 Ethylene C_2H_4	38.1 " "

The heating power would indicate 1582 B. T. U. per cubic foot, products condensed.

W. Ivison Macadam, F.C.S., *J. Soc. Chem. Industry*, March, 1887, tabulates the results of a series of his tests upon the Pintsch and Keith oil gas, as follows:

PARAFFIN OIL INTO GAS.

	Average of trials with Keith's apparatus.	Average of trials with Pintsch's apparatus.
Specific gravity of the oil	0.875	0.877
Weight of one gallon of the oil	8.758 lbs.	8.779 lbs.
Number of gallons per ton of oil	255.76	255.15
Flashing point	289° F.	295° F.
Burning point	347° F.	354° F.
Gas from one gallon of oil	84.93 c. ft.	90.03 c. ft.
" " " ton " "	21,720 c. ft.	24,757 c. ft.
Candle power of gas	61.38 candles.	60.82 candles.
Illuminating value of 1 cubic foot in grains of sperm	1473 grs.	1459 grs.
Illuminating value of 1 ton in lbs. of sperm	4570 lbs.	5160 lbs.

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[1] *Transactions Amer. Society Mechanical Engineers*, 14, (1892) 355.

	Average of trials with Keith's apparatus.	Average of trials with Pintsch's apparatus.
Illuminating value of 1 gallon in lbs. of sperm	17.876 lbs.	20.198 lbs.
Heavy hydrocarbons absorbed by bromine	39.05	38.20
Carbon dioxide	0.27	0.08
Dihydric sulphide	Decided.	None.

Oil gas, compressed to six or eight atmospheres, in iron cylinders, is extensively used for the lighting of railway carriages. When more pressure, say ten atmospheres, is used the gas loses hydrocarbons which settle out, and this loss in illuminants may cause twenty per cent. loss in the illuminating power of the gas.

References.—Gas Manufacture and Analysis. By W. J. Atkinson Butterfield, F.C.S., London, 1896.

Methods of Gas Analysis. By Dr. Walther Hempel, translated by Prof. L. M. Dennis, N. Y., 1892.

Oil Gas. By W. A. Noyes, W. M. Blink, and A. V. H. Mory, *J. Am. Chem. Soc.*, **16**, 688. (A report of a very complete test of an oil gas plant).

Technische Gasanalyse. By C. Winkler.

Chemisch-Technische Analyse. By Dr. Julian Post, Braunsweig, 1890.

NATURAL GAS.

The increased use of natural gas[1] in the metallurgical industries in the states of Pennsylvania, Ohio, and Indiana, has given this subject an enhanced value.

The composition of the gas is not uniform and consequently the heating power varies. Chemists are not in agreement with the statements of the results of analyses, as the following comparisons show :

ANALYSIS OF NATURAL GAS, BY DR. G. HAY, FOR THE NATURAL GAS COMMISSION.[2]

CO_2	0.00	per cent.
CO	1.00	" "
Heavy hydrocarbons	0.50	" "
CH_4	95.20	" "
H	2.00	" "
O	1.30	" "
N	0.00	" "
	100.00	" "

B. T. U. per cubic foot = 1036.87.

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[1] For the history of the development of natural gas in Pennsylvania, consult *Transactions Amer. Institute Mining Engineers*, 14, 423-439.

[2] *Engineering and Mining Journal*, 39, 247.

S. A. Ford (chemist to the Edgar Thomson Steel Works) reports analyses of natural gas, as follows:

	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	No. 6.	
CO_2....	0.80	0.60	0.00	0.40	0.00	0.30	per cent.
CO	1.00	0.80	0.58	0.40	1.00	0.60	" "
O......	1.10	0.80	0.78	0.80	2.10	1.20	" "
C_2H_4...	0.70	0.80	0.98	0.60	0.80	0.60	" "
C_2H_6...	3.60	5.50	7.92	12.30	5.20	4.80	" "
CH_4 ...	72.18	65.25	60.70	49.58	57.85	75.16	" "
H	22.02	26.16	29.03	35.92	9.60	14.45	" "
N......	0.00	0.00	0.00	0.00	23.41	2.89	" "
Total.	99.30	100.81	99.99	100.00	100.00	100.00	" "

Nos. 1–4 are analyses of gas from the same well, made at intervals of two months. Nos. 5 and 6 are from two different wells in the East Liberty District, Pa.

W. A. Noyes[1] gives the analysis of a sample of natural gas, from New Lisbon, Ohio, as follows:

CH_4..............................	67.00	per cent.
C_2H_6..............................	11.10	" "
CO_2..............................	1.20	" "
O..............................	0.90	" "
N..............................	19.80	" "
Total....................	100.00	" "

The number of B. T. U. per cubic foot amounting to 917.

The percentage of nitrogen in the gas being exceptionally high, the heating power is correspondingly reduced.

Another analysis (Taylor, *Trans. Amer. Inst. Mining Eng.*, **18**, 881) is reported as follows:

CO..............................	0.50	per cent.
H..............................	2.18	" "
CH_4..............................	92.60	" "
C_2H_4..............................	0.31	" "
CO_2..............................	0.26	" "
N..............................	3.61	" "
O..............................	0.34	" "
Total..................	99.80	" "

Each cubic foot containing 1000.52 B. T. U.

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[1] *Proceedings Amer. Asso. Advancement of Science*, 1893, p. 106.

The most complete investigation regarding the chemical composition of natural gas, has been made by Prof. F. C. Phillips for the Geological Survey of Pennsylvania.[1]

ANALYSIS OF FREDONIA NATURAL GAS. (PHILLIPS.)

N	9.54	per	cent.
CO_2	0.41	"	"
C_2H_4, etc	0.00	"	"
CO	0.00	"	"
Free hydrogen	0.00	"	"
NH_3	0.00	"	"
Hydrocarbons of paraffin series	90.00	"	"
Total	100.00	"	"

	Sheffield gas.	Wilcox gas.	Kane gas.		
N	9.06	9.41	9.79	per	cent.
CO_2	0.30	0.21	0.20	"	"
O	trace	trace	...	"	"
H	0.00	0.00	0.00	"	"
C_2H_4, etc	0.00	0.00	0.00	"	"
CO	0.00	0.00	0.00	"	"
Paraffins	90.64	90.38	90.01	"	"
Total	100.00	100.00	100.00	"	"

A practical test of the fuel value[2] of natural gas has been carried out by the Westinghouse Air-brake Co., of Pittsburgh, Pa. Taking the usual "best" quality of Pittsburg coal, it was found that its evaporating duty in a particular boiler was 10.38 pounds of water per pound of the solid fuel. With the same boiler 1.18 cubic feet of natural gas evaporated one pound of water; whence it follows that one pound of coal is equivalent to 12.25 cubic feet of gas, or that 1000 cubic feet of the gas were as good as $81\frac{31}{49}$ pounds of coal. According to calorimetric tests, 55.4 pounds of coal contain the same number of heat units as 1000 cubic feet of the natural gas.

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[1] Report on the Chemical Composition of Natural Gas," F. C. Phillips, *J. Franklin Institute*, 124, 242–256, 358–375.

[2] *Journal Iron and Steel Inst.*, 1887, 366-418. "Fuels," Mills and Rowan, p. 292.

XXXII.

Practical Photometry.

The illuminating value of any source of light is determined by comparing it with some source of light of known value. The illuminating value of gas is measured by comparing a flame that is burning at the rate of five cubic feet an hour with a standard sperm candle that is burning at the rate of 120 grains an hour.

The amount of light received by any object will vary inversely as the square of the distance of that object from the source of illumination, hence, if the light whose power is to be determined illuminates body at X inches to the same degree that a standard candle would illuminate that same body at Y inches, the illuminating power of that light will be $\frac{X^2}{Y^2}$ candles.

In constructing a photometer this single principle is kept in view, and all the refinements are to eliminate errors in judgment and to allow for the variations in the rate of combustion of gas and sperm.

The accompanying illustration shows the form of photometer known as the Bunsen, which is the one most commonly used in Germany, England, and America.

It consists first of a table which carries the apparatus and on which the distance between the lights is accurately laid off and marked by two lines. This distance is generally 60 inches, but 2 meters and 100 inches are also used. In case either light is changed or moved for any reason, it may easily be put back in place by placing it centrally over the line indicated on the table. To facilitate the adjustment two plumb-bobs are hung over each of the lines at the ends of the table, so it is easy to see whether the flames are properly centered in one direction. In the other direction they are centered by sighting along the bar. The bar is placed at right angles to the two lines laid out on the table and centrally between them. It is laid out in inverse squares so that "1" is in the center. If the length of the bar is Y and the distance from the candle is X, the candle-power is $\frac{(Y-X)^2}{X^2}$. The mark that indicates four candle-power is twice as far from the

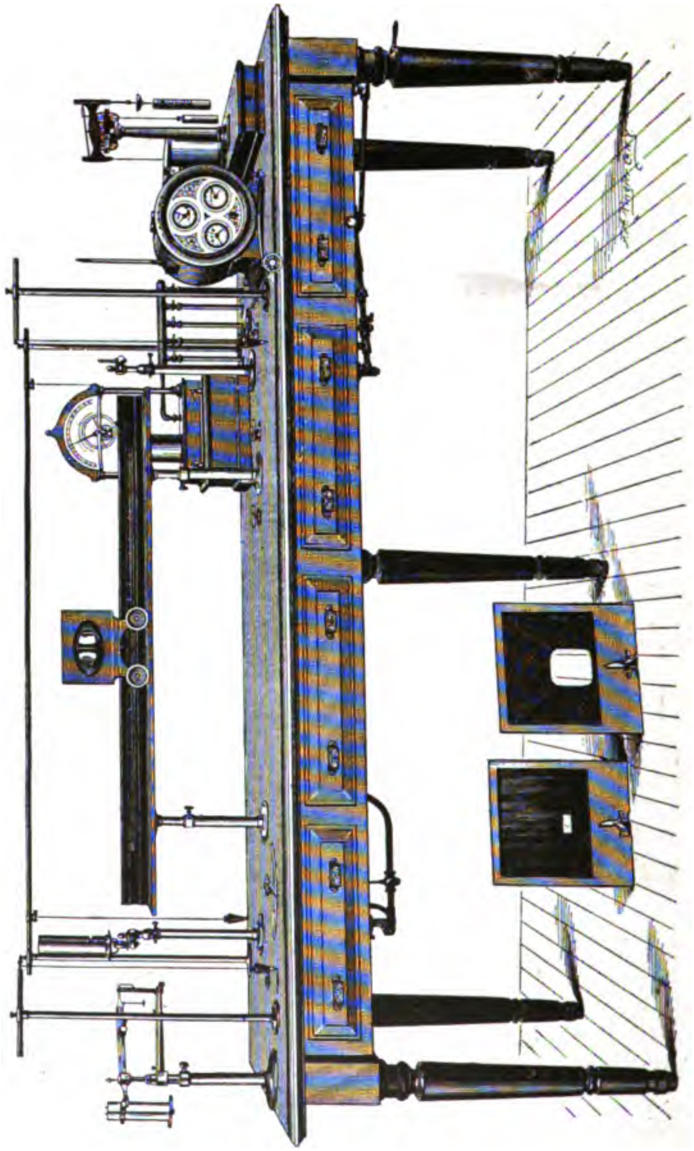

Fig. 85.

light to be measured as it is from the candle, 9 is three times as far, etc.

The bar should be made so that it may be raised or lowered at pleasure, and be planed to a thin edge on top so that no light will be reflected from it on the disk. On the bar is a sight-box in which a paper disk is placed at right angles to and centrally over the bar. There are several kinds of disks used, but the one most commonly preferred in this country is made by taking a piece of white sized paper of medium thickness, and cutting out of the center a many-pointed star about an inch and a half in diameter outside the points. This paper with the star cut from the center is then placed between two pieces of tissue paper and the three held together either by placing between pieces of glass or else by being fastened with thin starch water. At the back of the sight-box are two mirrors, so placed that the observer may stand in front of the bar and see both sides of the disk. On the front of the sight-box a hood is so placed as to partially screen the eyes of the observer from the lights.

At one end of the bar is the light to be tested. This is connected to a pipe sealed in mercury, so that it may be moved back and forth or raised and lowered at pleasure. It is usually arranged with a micrometer cock so that the rate of flow may be regulated as closely as may be necessary.

At the other end of the bar is a candle balance. The balance is usually arranged for two candles and all readings are multiplied by 2. This balance is so constructed that the position of the candles may be adjusted vertically or horizontally.

This end of the bar is so arranged that the candle balance may be removed and a standard burner put in its place. The standard burner commonly used is a Sugg Argand burner, size *D*. This is covered with a thin sheet metal chimney one and seven eights inches diameter. This chimney has an opening on one side $\frac{13}{32}$ inch high and one and one half inches wide. On the opposite side the chimney is cut away to prevent light being reflected through the slot in front. The standard burner, like the one through which the gas is tested, is so arranged that it may be adjusted in all directions.

A meter to measure the gas is necessary. As gas is burned at the rate of five feet an hour when being tested, the meter is so geared that one of the hands makes a complete revolution each time a twelfth of a foot of gas passes. A clock is attached to the meter with a large second hand, so when the meter hand mentioned and the second hand move together, gas is passing at the rate of 5 feet an hour. In addition to these hands are one indicating feet and one minutes. Some meters are furnished with a third set of hands reading feet and hundreds.

The meter has a thermometer to show the temperature of the gas and a universal level so that it may be properly leveled. On the side is a glass gauge and a mark indicating the height of the water, which should always be constant.

The pipe connections to the meter are so arranged that opening a cock will allow the gas to pass around instead of through it. This permits the operator to start or stop the meter at pleasure without interfering with the light.

A pressure gauge connected with the various parts of the apparatus enables the operator to ascertain the pressure of the gas at different points. One of these connections is to the pipe a short distance below the test burner. This gives the pressure near the point of ignition. The pressure is read in inches and fractions of an inch of water.

A gas governor is connected before the inlet to the meter, which reduces the pressure to about an inch and a half of water. Beyond the meter is a smaller governor which reduces the pressure to about nine-tenths of an inch and prevents alteration of the flow of gas due to the irregularities in the meter.

Black screens are arranged to screen the eye of the observer from the light. These are sometimes fixed and at others set on the bar. The latter arrangement is preferable, as they may be moved to suit different positions of the sight box.

For testing gas of not over eighteen candle power the Standard London Argand burner is used. For higher candle power gas the ordinary sawed lava tip is best. The latter is commonly known as the batwing burner.

The photometer should be set up in a small, light-proof room with dead black walls. The latter can be hung with black velvet or painted with glue and lampblack. Great care should be taken to insure proper ventilation without draft. The temperature of the room should be kept as near 60° F. as possible, and the air should not be allowed to become vitiated by the products of combustion. The table should be set so that readings may be taken from both sides of the bar.

MANNER OF USING THE PHOTOMETER.

When one starts to use a new photometer, or one with which the experimenter has not previously worked, the instrument should be carefully verified.

First, make sure that the lines defining the distance between the lights are the proper distance apart and parallel, and that the bar is perpendicular to and midway between them. Next see that the bar is level. The disk must be at right angles to the bar, and the small pointer under the sight box in line with the disk. The two mirrors should be made of the best plate glass and well silvered. They should be kept clean. The disk should exactly bisect the angle made by the mirrors. The bar should be verified so that the operator may be sure that it is properly divided, and the meter should be tested with a meter prover. In testing the meter be sure that the temperature of the room, of the water in the meter, and of the water in the prover are the same. The pressure gauge should be verified by a U-shaped water gauge. The knife edges of the candle balance should be clean and sharp, and the lever should be free to move without rubbing. The weight for the candle balance should be weighed on an analytical balance to be sure that it is correct.

For testing coal gas no choice is allowed in the burner, but when water gas or any high grade gas is to be tested it is necessary to get a burner suited to the gas. The most suitable burner can be quickly determined by experiment, and the greatest efficiency is usually obtained with a burner of such size that the gas is almost on the point of smoking. When the photometer light is burned continually, as is usually the case in gas works, the tip on the flat-flame burner should be changed at intervals of two or three weeks. Care should be taken that the tip is smooth. Any tips that are chipped on top or rough in the slot should not be used.

In preparing for a test, the burner and candles should be placed in their proper positions and at such a height that the center of the flames will be on a level with the center of the disk. The height of the candle flame is taken when the candle end of the balance is down. The gas should be burned long enough to be sure that the apparatus is cleaned out and that fresh gas is being burned. Before starting it is necessary to control the pressure under the burner so that it will not vary during the test. The governor on the outlet of the meter will do this if it is in order. If the pressure varies, the governor must be cleaned before starting the test. During the test the pressure gauge must be shut off, as in case there is change of pressure it will store or give out enough gas to vitiate the result. The meter should be level and the water at the proper height.

The wicks of the candles should never be touched. The candles are lighted and allowed to burn until the wick curls over to the edge of the flame and burns away as the candle is consumed. The end of the wick should glow. No test should be started until the wicks are bent over and the ends are glowing. The candles should always be burned eight or ten minutes before starting a test. A common practice which gives good results is to allow the candles to burn eight or ten minutes and then extinguish them for two or three minutes. The candles are then relighted and allowed to burn about two minutes before starting the test. They are commonly placed in the holders in such a way that the ends of the wicks are as far away from each other as possible.

When the apparatus has been brought to the proper condition for testing, the flow of gas is adjusted to as near five feet an hour as possible, and the meter is allowed to run until the twelfth of a foot hand points to 0, when it is by-passed. The clock is stopped at 0. The candles are counterbalanced by the sliding weight on the balance lever until the weight almost carries the lever down. In a few seconds the candles burn sufficiently to allow the balance to fall, and at that instant the meter and clock should be started. As soon after as possible the 40-grain weight should be dropped into the scale pan, which brings the candles down again. The operator should always move about the room deliberately so as to avoid as far as possible creating currents of air. The candle flames must be still before beginning to take readings.

A reading should be taken every minute for ten minutes. When the screen is apparently illuminated equally on both sides it should be moved a little to the right and to the left, and in each case the illumination on that side should increase. Five readings should be taken on one side of the bar and the sight-box turned around and five taken from the opposite side. In case the bar is accessible from only one side, the readings should be made with one eye and the screen turned in the sight-box after half have been completed. This will eliminate the errors due to possible difference in eyes and in the sides of the screen.

The last reading should be taken during the first half of the tenth minute and the times noted when the candle balance falls, and when the gas hand completes its tenth revolution. The temperature of the gas and the reading of barometer should also be noted. After this the candles may be extinguished. They should be blown out and the ends of the wicks extinguished with a piece of sperm. The wicks should never be touched with anything else.

If the candle balance falls in less than 9½ or more than 10½ minutes, or, if the gas hand takes less than 9½ or more than 10½ minutes to make 10 revolutions, the test should be discarded. Long practice has shown that within these limits the light given by the candles varies approximately with the consumption of sperm and that given by the burner approximately with the gas consumed.

If the candles take X seconds to burn 40 grains and the gas hand Y seconds to make 10 revolutions, the average reading multiplied by 2 should be multiplied by $\frac{X}{600} \times \frac{600}{Y}$ or $\frac{X}{Y}$. This will give the candle-power of the gas uncorrected for temperature and pressure.

The standard of pressure is 30 inches of mercury and the standard temperature is 60° Fahr. To correct the pressure multiply by 30 and divide by the barometric reading. In correcting for temperature the gas is assumed to be a perfect gas saturated with water-vapor. The following is the formula for correction for pressure and temperature:

$$n = \frac{17.64\ (h - a)}{460 + t}$$

n = the number by which the observed volume is to be multiplied to reduce it to 30 inches and 60°.

h = the height of the barometer in inches.

t = the temperature Fahrenheit.

a = the tension of aqueous vapor at $t°$.

The table on the opposite page will facilitate corrections for various pressures and temperatures.

Inasmuch as a flame is not perfectly transparent, a test made with it at right angles to the bar does not give the mean of the light that is emitted horizontally. The richer the gas the greater is the difference between the candle-power measured on the flat and on the edge of the flame. A gas that gives 25 candles measurement flat will not give over 19.5 candles measured on the edge. When the flame is at an angle of 10 degrees with the bar it gives almost as much light as when it is measured at 90 degrees.

The best photometers are made so that the burner may be turned on its axis and the light measured at all angles.

When it is desired to measure the light emitted by a burner at various altitudes, mirrors are used to reflect the light to the disk as the latter is kept vertical and in the same horizontal plane as the standard burner. In such cases it is necessary to test very carefully the amount of light absorbed by the mirrors at all angles.

There is a popular impression that photometrical work is not accurate and therefore not to be depended upon, but, if care is taken by the operator in his work, and all the apparatus is properly adjusted, the error will be less than 1 per cent. By taking the average of a series of measurements the error can be reduced to a point where it is inappreciable.

TABLE TO FACILITATE THE CORRECTION OF THE VOLUME OF GAS AT DIFFERENT TEMPERATURES AND UNDER DIFFERENT ATMOSPHERIC PRESSURES.

Barometer	Thermometer. F.															
	48°.	50°.	52°.	54°.	56°.	58°.	60°.	62°.	64°.	66°.	68°.	70°.	72°.	74°.	76°	78°.
28.0	0.960	0.956	0.951	0.946	0.942	0.937	0.932	0.927	0.922	0.917	0.912	0.907	0.902	0.897	0.892	0.887
28.1	0.964	0.959	0.955	0.951	0.945	0.941	0.936	0.930	0.926	0.921	0.916	0.911	0.905	0.900	0.895	0.890
28.2	0.967	0.963	0.958	0.953	0.949	0.944	0.939	0.934	0.929	0.924	0.919	0.914	0.909	0.904	0.898	0.893
28.3	0.971	0.966	0.961	0.957	0.952	0.947	0.942	0.937	0.932	0.928	0.922	0.917	0.912	0.907	0.902	0.896
28.4	0.974	0.970	0.965	0.960	0.955	0.951	0.946	0.941	0.936	0.931	0.926	0.921	0.915	0.910	0.905	0.900
28.5	0.978	0.973	0.968	0.964	0.959	0.954	0.949	0.944	0.939	0.934	0.929	0.924	0.919	0.914	0.908	0.903
28.6	0.981	0.977	0.972	0.967	0.962	0.958	0.953	0.947	0.943	0.938	0.932	0.927	0.922	0.917	0.912	0.906
28.7	0.985	0.980	0.975	0.970	0.966	0.961	0.956	0.951	0.946	0.941	0.936	0.931	0.925	0.920	0.915	0.909
28.8	0.988	0.984	0.979	0.974	0.969	0.964	0.959	0.954	0.949	0.944	0.939	0.934	0.929	0.924	0.918	0.913
28.9	0.992	0.987	0.982	0.977	0.973	0.968	0.963	0.958	0.953	0.948	0.942	0.937	0.932	0.927	0.921	0.916
29.0	0.995	0.990	0.986	0.981	0.976	0.971	0.966	0.961	0.956	0.951	0.946	0.941	0.935	0.930	0.925	0.919
29.1	0.999	0.994	0.989	0.984	0.979	0.975	0.969	0.964	0.959	0.954	0.949	0.944	0.939	0.933	0.928	0.923
29.2	1.002	0.997	0.992	0.988	0.982	0.978	0.973	0.968	0.963	0.958	0.952	0.947	0.942	0.937	0.931	0.926
29.3	1.006	1.001	0.996	0.991	0.986	0.981	0.976	0.971	0.966	0.961	0.956	0.950	0.945	0.940	0.935	0.929
29.4	1.009	1.004	0.999	0.995	0.990	0.985	0.980	0.975	0.969	0.964	0.959	0.954	0.949	0.943	0.938	0.932
29.5	1.013	1.008	1.003	0.998	0.993	0.988	0.983	0.978	0.973	0.968	0.962	0.957	0.952	0.947	0.941	0.936
29.6	1.016	1.011	1.006	1.001	0.996	0.992	0.986	0.981	0.976	0.971	0.966	0.960	0.955	0.950	0.944	0.939
29.7	1.019	1.015	1.010	1.005	1.000	0.995	0.990	0.985	0.980	0.974	0.969	0.964	0.959	0.953	0.948	0.942
29.8	1.023	1.018	1.013	1.008	1.003	0.998	0.993	0.988	0.983	0.978	0.972	0.967	0.962	0.957	0.951	0.946
29.9	1.026	1.022	1.017	1.012	1.007	1.002	0.997	0.991	0.986	0.981	0.976	0.970	0.965	0.960	0.954	0.949
30.0	1.030	1.025	1.020	1.015	1.010	1.005	1.000	0.995	0.990	0.985	0.979	0.974	0.968	0.963	0.958	0.952
30.1	1.033	1.029	1.024	1.019	1.014	1.009	1.003	0.998	0.993	0.988	0.983	0.977	0.972	0.966	0.961	0.955
30.2	1.037	1.032	1.027	1.022	1.017	1.012	1.007	1.002	0.996	0.991	0.986	0.980	0.975	0.970	0.964	0.959
30.3	1.040	1.036	1.030	1.025	1.020	1.015	1.010	1.005	1.000	0.995	0.989	0.984	0.978	0.973	0.968	0.962
30.4	1.044	1.039	1.034	1.029	1.024	1.019	1.014	1.008	1.003	0.998	0.993	0.987	0.982	0.976	0.971	0.965
30.5	1.047	1.042	1.037	1.032	1.027	1.022	1.017	1.012	1.006	1.001	0.996	0.990	0.985	0.980	0.974	0.969
30.6	1.051	1.046	1.041	1.036	1.031	1.026	1.020	1.015	1.010	1.005	0.999	0.994	0.988	0.983	0.977	0.972
30.7	1.054	1.049	1.044	1.039	1.034	1.029	1.024	1.018	1.013	1.008	1.003	0.997	0.992	0.986	0.981	0.975
30.8	1.058	1.053	1.048	1.043	1.037	1.032	1.027	1.022	1.017	1.011	1.006	1.000	0.995	0.990	0.984	0.978
30.9	1.061	1.056	1.051	1.046	1.041	1.036	1.031	1.025	1.020	1.015	1.009	1.004	0.998	0.993	0.987	0.982
31.0	1.065	1.060	1.055	1.049	1.044	1.039	1.034	1.029	1.023	1.018	1.013	1.007	1.002	0.996	0.991	0.985

XXXIII.

Hartley's Calorimeter for Combustible Gases.

The conditions of use are as follows: From a small cistern *A* (Fig. 86) water flows over a sensitive thermometer *B* thence into a case surrounding the stem of a suitable burner *C*, onwards to a metal casing or jacket enveloping the calorimeter *D*, then makes it way to the upper part of the latter, descends after traversing a series of shelves which present a very large surface, and finally passes out to the collecting tank *J*, at the base of the instrument. The burner is passed upwards into the center of a cylindrical chamber at the bottom of the calorimeter; and, as already stated, the burner stem is surrounded by a casing through

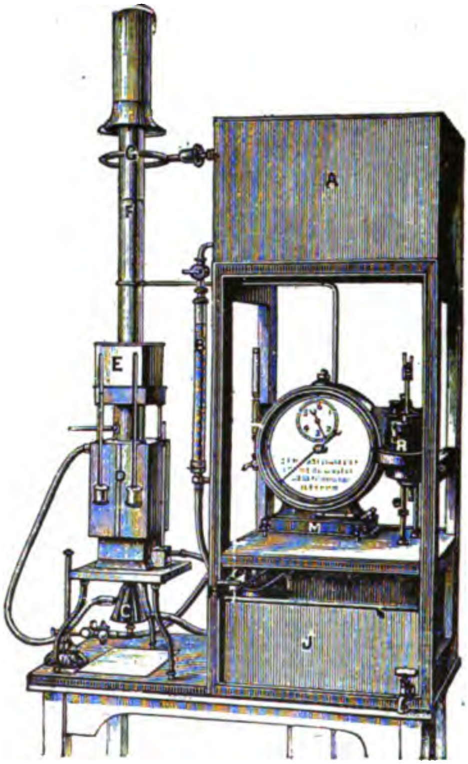

Fig. 86.

which the supply water flows. Loss by radiation from the burner is thus prevented.

The gas is measured by a special meter *M*: and its rate of consumption and the rate of water flow are regulated until the issuing water is found to be a few degrees higher in temperature than that at which it enters : and the temperatures, as indicated by the four thermometers employed, are found to be steady. During these adjustments, water runs to waste through a by-way cock. When all is ready, and with the meter index, the bye-way cock is instantly turned, and the passage of the outflowing water diverted to a collecting tank. The quantity of gas usually burned per experiment is one-fourth cubic foot; and the time occupied with ordinary coal gas ten to twelve minutes. During the experiment, the temperatures of the inlet and outlet water should be frequently observed, and now and then also the temperature of the jacket. When the desired quantity of gas has been burned, the water flow is promptly turned to waste.

The collected water is next measured, and its weight calculated, or better, weighed directly. The weight in pounds multiplied by the number of degrees the water has been raised gives the heating power due to the quantity of gas burned.

Thus, if twenty pounds of water have been raised 8° F, by one-fourth cubic foot of gas, we have $20 \times 8 = 160$ pounds, Fahr. units for one-fourth cubic foot, or 640 for one cubic foot.

Note.—The *products* of combustion are so completely reduced to the temperature of the inflowing water that, without aspiration, they would not rise through the instrument. The aspirator is simply a copper chimney *F*, heated at its upper part by a ring gas-burner *G*.

Prof. E. G. Love, *School of Mines Quarterly*, 13, 97, gives the result of an analysis, with this instrument, of a sample of the Municipal Gas Co. gas, of New York City, as follows:

Barometer	29.886	in.
Temperature of gas burned	66.00°	F.
Temperature of the air (a)	65.98°	"
Temperature of the water, inlet (b)	61.605°	"
Temperature of the water, outlet (c)	69.275°	"
Temperature of the water, raised	7.670°	"

Temperature of the "body" (d)	63.795° F.
Temperature of the escaping gases	64.43° "
Duration of test	12.78 minutes.
Gas burned	0.25 cubic feet
Gas burned, corrected to 60° F. and 30 in Bar	0.2452 " "
Pounds of water heated	23.228 " "
Corrections	

$(a-d)\ 2.185 \times 0.025 \times 1278 = 0.698°$ gain.
$(c-a)\ 3.295 \times 0.01 \times 12.78 = 0.421°$ loss.

$0.2770°$ gain.

$23.228 + 7.67 - 0.28 = 177.88 \div 0.2452 = 725.3$ heat units at 60° F. and thirty inches barometer.

The coal gas of London, Eng., with an illuminating power of sixteen to seventeen candles, has a colorific power of about 668 B. T. U. per cubic foot, and costs from sixty to seventy cents per thousand cubic feet.

The average of numerous tests, made with the Hartley calorimeter, upon the New York City water gas, gives 710.5 B. T. U. per cubic foot. One thousand cubic feet of this gas, costing $1.25, would therefore yield 710,500 heat-units, which would be equivalent to 568,400 B. T. U. for $1.00.

XXXIV.

Junker's Gas Calorimeter.

Another form of gas calorimeter is the Junker, (Fig. 87).[1] The above sectional drawing shows the instrument to consist

1. Cold water inlet.
2. Strainer.
3. Overflow to calorimeter.
4. Upper container.
5. Waste overflow.
6 and 7. Fall pipe and joint.
8. Drain cock.
9. Adjustment cock.
12. Cold water thermometer.
13. Air jacket.
14. Perforated spreading ring.
15 and 16. Water jacket.
17. Baffle plates with cross slots.
18. Lower overflow.
19. Lower container.
20. Hot water overflow.
22. Gas nipple.
23. Air supply regulator.
24. Gas nozzle.
25. Clamp for burner.
26. Burner holder.
27. Burning cap.
28. Combustion chamber.
29. Roof of combustion chamber.
30. Cooling tubes.
31. Receiver for combustion gases.
32. Outlet for combustion gases.
33. Throttle for " "
34. Brass base ring.
35. Condensed water outlet.
36. }
37. } Air jacket.
38. }
39. Test hole in air jacket.
43. Hot water thermometer,

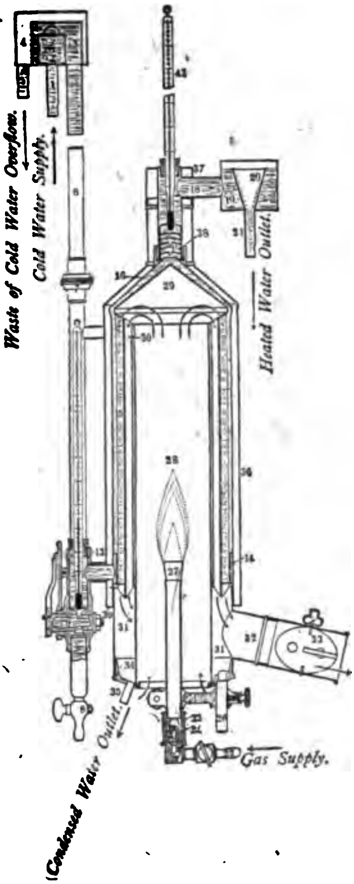

Fig. 87.

of a combustion chamber surrounded by a water jacket, the latter filled with a great many tubes. To prevent loss by radiation

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[1] *J. Soc. Chem. Ind. July, 1895*, 632.

the water jacket is surrounded by a closed air space. The whole apparatus is constructed of copper as thin as is compatable with strength. The water enters the water jacket at the bottom, and leaves it at the top, while the hot combustion gases of the flame of the gas that is on trial enter the tubes at the top and leave them at the bottom. There is therefore not only a very large surface of thin copper between the gases and the water, but the two move in opposite dire[illegible], during which process all the heat generated by the flame [illegible] transferred to the water, and the water gases leave the apparatus approximately at atmospheric temperature. The gas to be burned is first passed through a meter, and then to insure constant pressure, through a pressure regulator. The source of heat in relation to the unit of time is thus rendered stationary, and, in order to make the absorbing quantity of heat also stationary, two overflows are provided at the calorimeter, making the head of the water and the rate of flow of the same constant. The temperatures of the water entering and leaving the appratus can be read at the respective thermometers; as shown before, the quantities of heat and water passed through the apparatus are constant. As soon as the flame is lighted the temperature of the exit thermometer will rise to a certain point and will nearly remain there. All data for ascertaining the heat given out by the flame are therefore available.

All that is required is to measure simultaneously the quantity of gas burned and the quantity of water pressed, and the difference in temperature between the entering and leaving water. Centigrade thermometers and two-liter flasks are required.

The meter shows one-tenth of a cubic foot per revolution of the large hand; the circumference being divided into 100 parts, so that 0.001 can be read accurately. The water supply is so regulated that the overflow is working freely, and the water-admission cock is set to allow two liters of water to pass in about a minute and a half. The colorimeter is now ready to take the reading. The cold water as a rule has a sufficiently constant temperature that we note it only once: it is now 17.2° C. As soon as the large index of the meter passes zero, note the state of the meter and at the same time transfer the hot-water tube from the funnel into the measure glass, and while that is being filled note the temperature of the hot water at say ten intervals, to draw the average.

The temperatures are 43.8, 43.5, 43.5, 44.2, 44.1, 43.9, 43.8, 43.7, 43.8, and 43.7, making the average 43.8.

The measure glass is now filled; turn the gas out. Find from the readings of the meter at the beginning and the end of the experiment that there was burned 0.35 cubic foot, by means of which the temperature of the two liters of water was raised 26.6° C., *viz.*, 43.8°—17.2° = 26.6° C. The calculation is as follows:

$$H = \frac{W\,T}{G},$$

where $H =$ the calorific value of one cubic foot of gas in calories.

$W =$ the quantity in liters of the water heated.

$T =$ the difference in temperature between the two thermometers in degrees C., and $G =$ the quantity in cubic feet of gas used, then

$$H = \frac{2 \times 26.6}{0.35} = 152 \text{ calories or } 604\ (152 \times 3.968)\ \text{“B. T. U.”}$$

per cubic foot.

It is mentioned before that the effect of the cooling water is such that the waste gases leave the calorimeter at about atmospheric temperature. All hydrocarbons when burned form a considerable quantity of water, which in all industrial processes escapes with the waste gases as steam. The latent heat of this steam is therefore not utilized when fireing a stove or driving an engine with gas; in the above result, however, the latent heat *is* included, because in the copper tubes the steam is condensed, and its heat is transferred to the circulating water and measured with the rest. The condensed water runs down the tubes which are cut off obliquely to allow the drops to fall off easily, and is collected in the lower part of the apparatus from where it runs through the little tube into a measure glass. In condensing steam gives off six-tenths calorie for every cubic centimeter of water formed. If therefore a graduated (cc.) cylinder be placed under the little tube the amount of water generated by burning say one cubic foot of gas, can be directly measured.

From burning one cubic foot of gas, we have collected 27.25 cc.

of condensed water, and must therefore deduct 16.35 calories from the gross value found above, which gives the net calorific value of the gas tested as 135.65 calories or 538 B. T. U. per cubic foot.

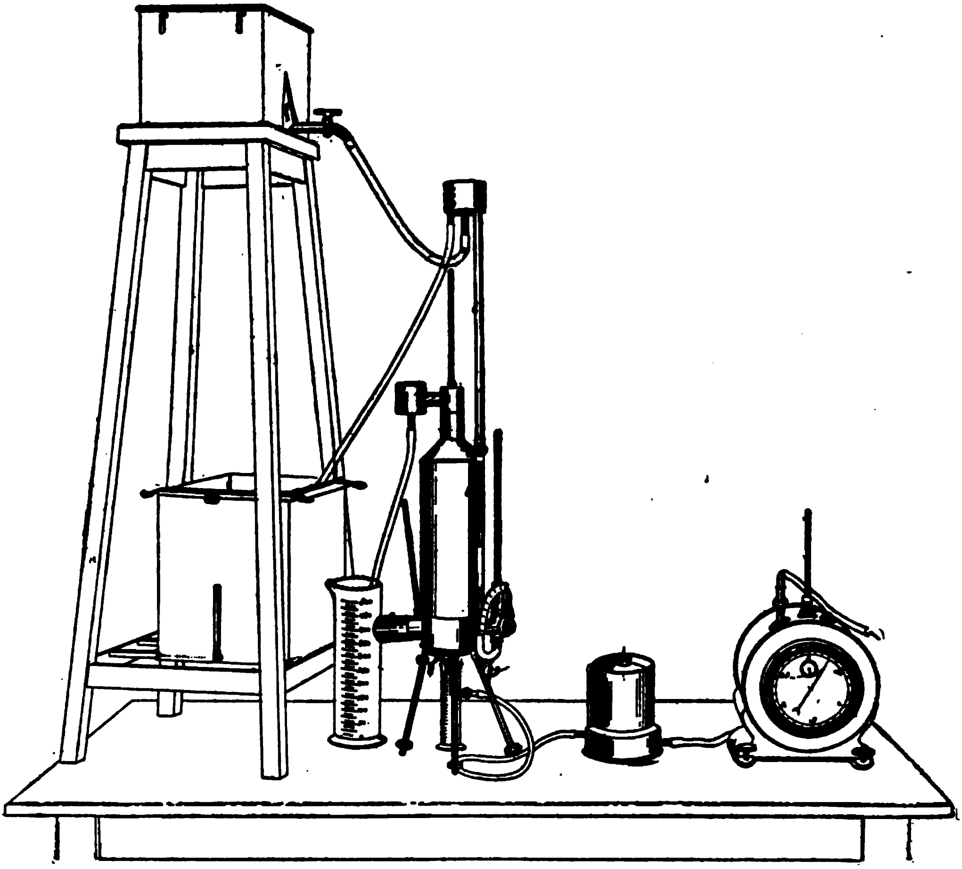

Fig. 88.

The calorimeter is placed so that one operator can simultaneously observe the two thermometers of the entering and escaping water, the index of the gas-meter, and the measuring glasses.

No draught of air must be permitted to strike the exhaust of the spent gas.

The water supply tube is connected to the nipple in the center of the upper container; the other nipple is provided with a waste tube to carry away the overflow. This overflow must be kept running while the readings are being taken.

The nipple, through which the heated water leaves the calorimeter, is connected by an india-rubber pipe with the large measure glass, and the water must be there collected without splashing.

The smaller measure glass is placed under the tube to collect any condensed water.

After the thermometers have been placed in position with their india-rubber plugs, the water supply is turned on by the cock, and the calorimeter filled with water until it begins to discharge. No water must at this period exude from the smaller pipe or from the test hole under the air jacket, otherwise this would prove the calorimeter to be leaking.

TABLE OF RESUMÊ OF TESTS UPON LONDON COAL GAS.

	Temperature of room.	Temperature of water inlet.	Temperature of water outlet.	Rise in temperature.	Gas burned. Cubic feet per minute.	Temperature of products of combustion.	Condensed water per cubic foot of gas.	Gross calories per cubic foot.	Latent heat of steam.	Net calories per cubic foot.
First day..	21.0°	15.322	26.113	10.79	0.0407	...	25.7	165.3	15.4	149.9
Second " ..	22.5°	12.9	27.68	14.78	0.0584	...	27.4	165.9	16.4	148.5
Third " ..	17.5^{C}	13.71	28.6	14.89	0.1103	17.5	26.43	164.8	15.86	148.94
Fourth " ..	17.5^{C}	13.75	28.53	14.78	0.1103	17.4	26.43	165.6	15.86	149.74

Experiments made with this calorimeter at the Stevens Institute, are recorded in the *Stevens Indicator*, October, 1896.

The gas used was carburetted water gas "Lowe Process" composed as follows:

CO_2	2.20	per cent. (by volume).
Illuminants $\left\{\begin{matrix} C_2H_4 \\ C_3H_6 \\ C_6H_6 \end{matrix}\right\}$	12.80	" " " "
O	0.00	" " " "
CO	24.20	" " " "
CH_4	17.83	" " " "
H	37.95	" " " "
N	5.02	" " " "
	100.00	

The theoretical heating value of this gas, is 662 B. T. U. per cubic foot.

The heating value as determined with the Junker calorimeter is 668 B. T. U. per cubic foot.

XXXV.

Liquid Fuel.

Petroleum containing eighty-six per cent. of carbon has an evaporative power, as estimated by Storer, of eighteen pounds of water per pound of petroleum. Deville has determined the heating power of various petroleums, by calorimetric tests, with the following results:

Heavy oil from West Virginia	10180	calories per kilo.
Light " " " "...........	10223	" " "
Heavy " " Ohio.................	10399	" " "
Light " " Penn.................	9963	" " "
Petroleum from Java.................	10831	" " "
Petroleum from Alsace	10458	" " "
Petroleum from E. Galacia..........	10005	" " "
Petroleum from W. Galacia	10235	" " "
Crude shale oil from Autun (France)	9950	" " "

Dr. Paul estimates the evaporative power of liquid hydrocarbons as the sum of the carbon and hydrogen present, on the basis that when oxidized with the theoretical proportion of air, each pound of carbon evaporates 11.359 pounds of water at 15.5° C., and each pound of hydrogen 41.895 pounds of water at 15.5° C., into steam at 100° C. The following table gives the results obtained :

	Carbon.	Hydrogen.	Oxygen.	Evaporation power in pounds of water at 100° C.	Evaporation duty in pounds of water at 15.5° C.
C_6H_6O (Phenol)....	76.6	6.49	17.00	12.24	10.50
C_7H_8O (Cresol)	77.77	7.41	14.82	13.00	11.16
$C_{10}H_8$ (Naphthalin).	93.75	6.25	....	15.43	13.07
$C_{14}H_{10}$ (Anthracine)	94.38	5.62	....	15.24	13.26
C_8H_{10} (Xylol)......	90.56	9.44	....	16.58	14.24
C_9H_{12} (Cumol)	90.00	10.00	....	16.78	14.41
$C_{10}H_{14}$ (Cymol).....	89.55	10.45	....	16.94	14.55

The *effective* heat he calculates as follows, using twice the amount of air required by theory for the combustion.

Combustion of One Pound of Carbon.

	B. T. U. Heat units.	Equivalent evaporation of water. At 100° C.	At 15.5° C.
Total heat of combustion	14500	15.0 lbs.	.. lbs.
Available heat	14500	... "	.. "
Waste of furnace gases at 315° C.	3480	3.6 "	.. "
Effective heat	11020	11.4 "	9.8 "

Combustion of One Pound of Hydrogen.

	B. T. U.	Equivalent evaporation of water. At 100° C.	At 15.5° C.
Total heat of combustion	62032	64.2 lbs.	.. lbs.
Latent heat of water vapor	8695	... "	.. "
Available heat	53337		
Waste heat of furnace gases	11520	11.9 ,,	.. "
Effective heat	41817	43.3 "	38 "

The *effective* heat of two hydrocarbons (containing respectively carbon eighty-six per cent., hydrogen fourteen per cent., and carbon seventy-five per cent., hydrogen twenty-five per cent.) are thus tabulated :

Hydrogen Containing Carbon Eighty-Six Per Cent., Hydrogen Fourteen Per Cent.

		Total heat of combustion.	Equivalent evaporation of water. At 100° C.	At 15.5° C.
C = 0.86 × 14500		12470 B. T. U.	......	......
H = 0.14 × 62032		8684 "	......	......
		21154	21.9 lbs.	18.8 lbs.
Furnace gases.		Heat units in furnace gases.		
CO_2	3.16 lbs.	411 B. T. U.	......	......
Water vapor	1.26 "	359 "	......	......
N	11.45 "	1683 "	......	......
Surplus air	14.37 "	2124 "	2.2 lbs.	......
	30.24 "	4577 "	4.8 "	
Total heat of combustion		21154 "	......	......
Latent heat of water vapor		1217 "	1,3 lbs.	......
Available heat		19937 "		
Waste in furnace gases		4577 "	4.8 "	......
Effective heat		15360 "	15.8 "	13.6 lbs.
Theoretical evaporating power			21.9 "	

Hydrocarbon Containing Carbon Seventy-Five Per Cent., Hydrogen Twenty-Five Per Cent.

		Total heat of combustion.	Equivalent evaporation of water.	
			At 100° C.	At 15.5° C.
C = 0.75 × 14500		10775 B. T. U.	......	......
H = 0.25 × 62032		15508 "	......	......
		26283 "	27.1 lbs.	23.1 lbs.
Furnace gases.		Heat units in furnace gases.		
CO_2	2.75 lbs.	358 B. T. U.	.....	......
Water vapor	2.25 "	641 "	......	......
N	13.39 "	1968 "	......	......
Surplus air	17.39 "	2483 "	2.6 lbs.	......
	35.78	5450		
Total heat of combustion		26283 "	......	......
Latent heat of water vapor		2174 "	2.2 lbs.	......
Available heat		24109 "		
Waste in furnace gases		5450 "	5.6 "	......
Effective heat		18659 "	19.3 "	16.5 lbs.
Theoretical evaporating power			27.1 "	

The *theoretical evaporative efficiency* of different combustibles is estimated by Rankine from their chemical composition as follows:

$E = 15C + 64H - 8O$, and to calculate the quantity of air required for combustion, $A = 12C + 36H - 4\frac{1}{2}O$, from which the following table is derived.

Description of fuel.	Chemical composition. C.	H.	O.	A.	E.	Evaporation due to C.	$H - \frac{O}{8}$
Charcoal	0.93	0.00	0.00	11.5	14.0	14.0	0.00
Coke	0.88	0.00	0.00	10.6	13.2	13.2	0.00
Rock Oils $C_{18}H_{20}$	0.84	0.16	0.00	15.75	22.7	12.7	10.00
Rock Oils $C_{26}H_{28}$	0.85	0.15	0.00	15.65	22.5	12.66	9.84
Coal	0.87	0.05	0.04	12.0	15.9	13.02	2.85
Coal	0.75	0.05	0.05	10.6	14.1	11.25	2.85
Ethylene, C_2H_4	0.75	0.25	0.00	18.8	27.3	11.25	16.05
Acetylene, C_4H_2	0.85	0.14	0.00	15.43	22.1	12.9	9.2
Peat, dry	0.56	0.06	0.31	7.7	10.0	8.5	1.5
Wood, dry	0.58	0.05	0.40	6.0	7.5	7.5	0.00

Rankine adopts as his unit, the weight of fuel required to evaporate one pound of water at 100° C. under a pressure of 14.7 pounds per square inch this being equivalent to 966 B. T. U. The results were reduced as follows:

Let E be the corrected and reduced evaporation.

$e =$ the weight of water evaporated.

$T_1 =$ the standard boiling point (212 F.).

$T_f =$ the temperature of the feed water.

$T_b =$ the actual boiling point observed: then

$$E = e \left\{ 1 + \frac{T_1 - T_f + 0.3\,(T_b - T_1)}{966 \text{ F.}} \right.$$

This represents the number of times its own weight of water that a fuel would evaporate if there were no waste of heat, as however there is always a loss of heat, the efficiency of a furnance would be $\frac{E(\text{available})}{E\ (\text{total})}$.

The loss of units of evaporation by waste gases Rankine gives;

Loss by chimney $= \frac{1 + A'}{4000}\ T_c\ (\text{F}^\circ.)$

where $1 + A'$ equals the weight of burnt gas per unit of fuel and T_c (F) the temperature of the chimney gases above that of the atmosphere.

For ordinary coal $1 + A'$ ranges from thirteen to twenty-five, and hydrocarbon oils it is 16.3 if no excess of air is necessary above what is required for the combustion of the fuel.

Rankine gives the theoretical evaporative power of hydrogen and carbon as follows:

	Oxygen per unit of weight.	Air per units of weight.	Units evaporated.
H	8	36	64.2
Carbon, solid (charcoal)	2⅔	12	15.0
Carbon gas in 2⅓ parts CO	1½	6	10.5
Carbon, gaseous	2⅔	12	21.0

In 1892, from tests made for the Engineer's Club of Philadelphia, the relative heating value of coal, gas and petroleum are thus stated:

	Lbs. of water, from and at 212° F.
1 lb. anthracite coal evaporated	9.7
1 " bituminous " "	10.14
1 " oil 36^C B	16.48
1 cubic foot gas, 20 C. P	1.28

E. C. Potter, (Trans. Am. Inst. Mining Engineers, Vol. xvii, p. 807), states results of tests, at South Chicago Steel Works, of heating value of petroleum and block coal, as follows:

With coal, fourteen tubular boilers, sixteen feet by five feet, required twenty-five men to operate them: with fuel oil, six men were required, a saving of nineteen men at $2.00 per day or $38.00 per day. For one week's work 2,731 barrels of oil were used, against 848 tons of coal required for the same work. With oil at sixty cents per barrel and coal at $2.15 per ton, the relative cost of oil to coal is as $1.93 to $2.15.

XXXVI.

Valuation of Coal for the Production of Gas.

Take 100 grams of the coal in small lumps, so that they may be readily introduced into a rather wide combustion tube. This is drawn out at its open end (after the coal has been put in) so as to form a narrow tube, which is to be bent at right angles; this narrower open end is to be placed in a wider glass tube, fitted tight into a cork fastened into the neck of a somewhat wide-mouthed bottle serving as tar vessel. The cork alluded to is perforated with another opening wherein is fixed a glass tube bent at right angles, for conveying the gas, first through a calcium chloride tube, next through Liebig's potash bulbs containing a solution of caustic potash, having lead oxide dissolved in it. Next follows another tube partially filled with dry caustic potash and partly with calcium chloride; from this last tube a gas-delivery tube leads to a graduated glass jar standing over a pneumatic trough, and acting as gas-holder. Before the ignition of the tube containing the coal is proceeded with, all the portions of the apparatus are carefully weighed and next joined by means of india-rubber tubing. After the combustion is finished, which should be carefully conducted so as to prevent the bursting or blowing out of the tube, the different pieces of the apparatus are disconnected and weighed again. The combustion tube has to be weighed with the coal after it has been drawn out at its open end, and with the coke after the end of the combustion when it is again cold, and for that reason care is required in managing it. We thus get the quantity of coke, tar, ammoniacal water, carbon dioxide and hydrogen sulphide (as lead sulphide), and the gas is measured by immersing the jar in water, causing it to be at the same level inside and out.

Empty the Liebig's bulbs into a beaker and separate the lead sulphide by filtration, wash well, dry and weigh. From the lead sulphide the hydrogen sulphide present is calculated. This process, devised by the late Dr. T. Richardson, of Newcastle-on-Tyne, was found by him to yield very reliable results, so as to be suitable for stating what quantity of gas a ton of coal thus analyzed would yield.[1]

Newbigging's Experimental Plant for the Determination of the Gas-Producing Qualities of Coal.

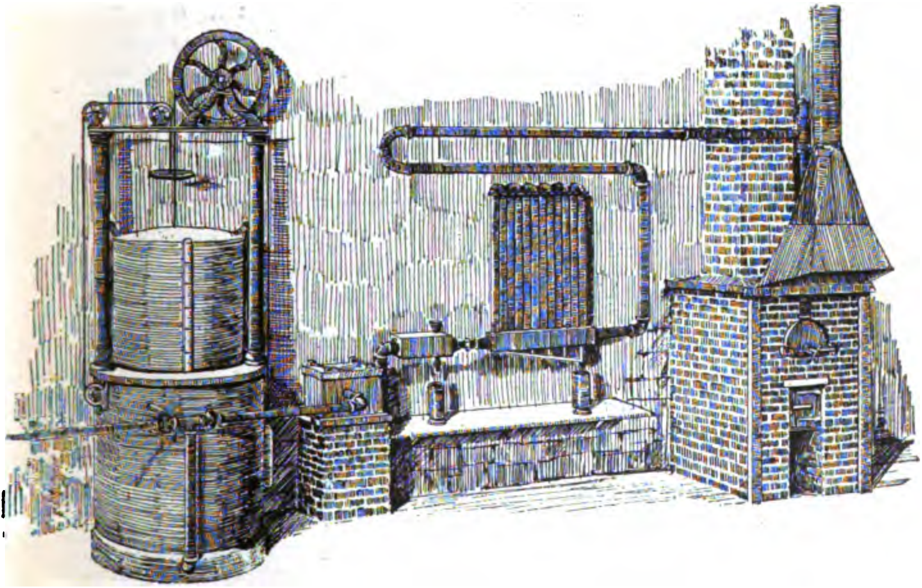

Fig. 89.

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[1] Crookes' Select Methods in Chemical Analysis, p. 607.

A description of the apparatus and method of use are thus given:

Retort—Cast iron: five inches wide, four and one-half inches high, two feet three inches long outside, and one-half inch thick.

Ascension pipe—Two inch wrought tube.

Connections—One and one-half inch wrought tube.

Condenser—Twelve vertical, one and one-half inch wrought tubes, each three feet six inches long.

Washer—One foot long, six inches wide, six inches deep.

Purifier—One foot two inches square, twelve inches deep, with two trays of lime.

Gas-holder—Capacity twelve cubic feet, with graduated scale attached.

Amount of coal to be taken for each test is $\frac{1}{1000}$ part of a ton, or 2.24 pounds. Care should be taken to obtain a fair average sample of the coal to be operated upon. For that purpose at least fifty pounds of coal should be broken up into small pieces and thoroughly intermixed, and from this three different charges are to be taken. The retort should be at a bright red heat before the introduction of the coal and maintained at that temperature during test. If from any cause the temperature is much reduced, the test will not be satisfactory. The time required to work off the charge of 2.24 pounds will range from forty to sixty minutes, according to the character of the coal. The illuminating power of the gas given out from each charge should be ascertained by the Bunsen photometer, no other being sufficiently trustworthy for that purpose. The average of the three is then taken, both for yield of gas and coke and for the illuminating power of the gas, and this fairly represents the capabilities of the coal. The further conditions to be observed are that the holder be emptied of air or of the previous charge of gas, and that the condenser be drained of its contents. The test charge may be continued until the whole of the gas is expelled, or otherwise, depending on circumstances. In comparing two coals, an equal production from both may be obtained, and the comparative illuminating power then ascertained.

The coke and "breeze" should be carefully drawn from the retort into a water-tight receptacle made of sheet iron closed by a lid. This is then placed in a bucket or other vessel of cold water, and when sufficiently cooled, the coke is weighed.

For ascertaining the quantity of tar and ammoniacal liquor produced, drain the yield of three charges from the condenser and washer and measure this in a graduated liquid measure. The number of fluid minims in a gallon (English) is 76,800. Then

Pounds. **Pounds per ton.**

$$6.75\left\{\begin{matrix}\text{The weight of}\\ \text{three charges}\\ \text{of coal.}\end{matrix}\right\} : 2240 :: \left\{\begin{matrix}\text{The number of}\\ \text{minims of tar}\\ \text{and liquor ob-}\\ \text{tained.}\end{matrix}\right\} : \left\{\begin{matrix}\text{The total num-}\\ \text{ber of minims}\\ \text{of tar and liquor}\\ \text{from a ton of}\\ \text{coal.}\end{matrix}\right\}$$

and this amount divided by 76,800 gives the gallons of tar and liquor produced per ton.[1] A good variety of gas coal should produce from 2,240 pounds of coal 12,000 cubic feet of gas, illuminating power twenty sperm candles.

Newcastle coal on an average produces 12,700 cubic feet of gas per ton of coal, illuminating power of fifteen sperm candles.

XXXVII.

Analysis of Clay, Kaolin, Fire Sand, Building Stones, Etc.

To be Determined.—Silica, (total), (combined), (free), (hydrated), alumina, lime, magnesia, potash, soda, ferrous or ferric oxide, manganous oxide, titanic oxide, sulphur trioxide and combined water.[2]

The total silica is determined by fusing one gram of the clay (previously dried at 100° C.) with ten parts of an equal mixture of sodium and potassium carbonates, in a large platinum crucible. Fusion must be complete and maintained at a red heat thirty minutes.

Allow to cool, treat with an excess of boiling water, make acid with hydrochloric acid, transfer solution to a four-inch porcelain capsule and evaporate to dryness. Take up with twenty-five cc. hydrochloric acid, add water, boil and filter upon ashless filter. Wash well with boiling water, dry, ignite and weigh as silica (total).

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[1] Newbigging's Handbook for Gas Engineers, p. 57.

[2] For analysis of limestone consult Scheme xi, page 16.

The forms of combination of the silica in the clay are determined as follows:[1]

Let A represent silica in combination with bases of the clay.

Let B represent hydrated silicic acid.

Let C represent quartz sand.

Dry two grams of the clay at a temperature of 100° C., heat with sulphuric acid, to which a little water has been added, for eight or ten hours, evaporate to dryness, cool, add water, filter out the undissolved residue, wash, dry and weigh $A+B+C$. Now transfer it in small portions at a time to a boiling solution of sodium carbonate (1 : 10) contained in a platinum dish, boil for some time, filter off each time, still very hot. When all is transferred to the dish, boil repeatedly with strong solution of sodium carbonate, until a few drops of the fluid, passing through the filter, finally remains clear on warming with ammonium chloride. Wash the residue, first with hot water, then (to ensure the removal of every trace of sodium carbonate which may still adhere to it) with water slightly acidified with hydrochloric acid, and finally with water. This will dissolve $A+B$, and leave a residue C of sand, which dry, ignite and weigh.

To determine B boil four or five grams of the clay (previously dried at 100° C.) directly with a strong solution of sodium carbonate, in a platinum dish as above, filter and wash thoroughly with hot water. Acidify the filtrate with hydrochloric acid, evaporate to dryness and determine this silica. It represents B or the hydrated silicic acid. Add together the weights of B and C thus found and subtract the sum from the weight of the first residue $A+B+C$. The difference will be the weight of A or silica in combination with bases in the clay.

If the weight of $A+B+C$ found here be the same as that of the silica found by fusion, in another sample of the clay of the same amount, the sand is quartz, but if the weight of $A+B+C$ be greater, then the sand contains silicates.

The weight of the bases combined with silica to silicates can be found by subtracting the weight of total silica found in one gram, by fusion, from the weight of $A+B+C$ in one gram.

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[1] From Fresenius, Quant. Anal., Cairn's, p. 68.

Alumina, Ferric Oxide, Manganese Dioxide, Lime and Magnesia.

The hydrochloric acid filtrate from the silica (by fusion) is made nearly alkaline with sodium carbonate, then excess of sodium acetate added, the solution boiled five minutes, then filtered by decantation and washed well.

<table>
<tr>
<td rowspan="3">Residue, $Al_2O_3.Fe_2O_3$.
Dissolve in HCl and divide into two equal portions.
<i>First Portion.</i>—Make alkaline with NH_4OH, boil and filter, wash, dry, ignite and weigh as $Al_2O_3.Fe_2O_3$.
<i>Second Portion.</i> — Titrate for iron (see Scheme XIII), calculate Fe found to Fe_2O_3, and this subtracted from weight of $Al_2O_3.Fe_2O_3$ gives weight of the Al_2O_3.
Both weights to be multiplied by 2.</td>
<td colspan="3">Filtrate.
Transfer to a flask, add a few drops of Br, set aside twelve hours (consult Scheme XIII), filter and wash.</td>
</tr>
<tr>
<td rowspan="2">Residue, MnO_2.
Dry, ignite and weigh as Mn_3O_4.</td>
<td colspan="2">Filtrate.
Add a few drops of ammonia (reaction of solution must be alkaline), then an excess of solution of ammonium oxalate, set aside four hours, filter and wash.</td>
</tr>
<tr>
<td>Residue, CaC_2O_4.
Dry, ignite and weigh as CaO.</td>
<td>Filtrate.
Add solution of sodium phosphate with stirring, set aside four hours, filter, wash with ammoniacal water, dry, ignite and weigh as $Mg_2P_2O_7$, and calculate to MgO.</td>
</tr>
</table>

Potash and Soda.

Take one gram of the dried clay, transfer to a three-inch platinum capsule, add ten cc. sulphuric acid and twenty cc. hydrofluoric acid and heat gently until the silica is completely dissipated and the excess of acid added driven off. Allow to cool, add twenty cc. warm hydrochloric acid, then twenty-five cc. water, transfer contents of platinum capsule to a No. 3 beaker, add two cc. nitric acid and boil. Add ammonia to alkaline reaction, boil, filter off the alumina and ferric oxide, and to the filtrate add ammonium oxalate to precipitate the lime; allow to stand four hours, then filter; the magnesia is separated in the filtrate by ammonium phosphate, and the filtrate from the magnesium phosphate precipitate is evaporated to dryness and ignited to expel ammonium salts. The residue is treated with hydrochloric acid and the potash precipitated by solution of platinic chloride as usual, and weighed as K_2PtCl_6 on counterpoised filters. The alcoholic washings and filtrate is evaporated to dryness, the platinum compound decomposed by heating to redness with oxalic acid, boiled with water, filtered, a few drops of sulphuric acid added, then evaporated to dryness, ignited to constant weight as sodium sulphate, and then calculated to Na_2O.

Sulphur Trioxide

Is determined by fusing one gram of the clay with sodium and potassium carbonates, separating the silica as usual, and precipitating the sulphur trioxide by solution of barium chloride in the acid filtrate. (Consult Scheme XIII).

Titanic Oxide.

Fuse five grams of the dried clay with an excess of a mixture of sodium fluoride and sodium bisulphate, in a platinum crucible for thirty minutes at a red heat. Treat the cold mass with cold water, about 200 cc., add potassium hydroxide in excess, filter off the titanic oxide, wash, dry and ignite and fuse this titanic oxide with about twelve times its weight of acid sodium sulphate; allow to cool, and treat with concentrated sulphuric acid. This is now added to 600 cc. of water, boiled for one hour, and the precipitated titanic oxide filtered, dried and weighed. (Consult Scheme XIII, Determination of Titanium).

Water of Hydration.

Take two grams of the clay, dried at 100° C., transfer to a covered platinum crucible and ignite over a blast-lamp at a red heat to constant weight. The loss represents the combined water.

Clays or fire sands that are to be used in the manufacture of fire bricks, retorts, etc., should contain only small amounts of easily fusible materials, such as potash, soda or iron; less than one per cent. of either alkali, or two per cent. of iron oxide being allowable in the best fire clays.

COMPOSITION OF SOME REPRESENTATIVE FIRE CLAYS.

	1.	2.	3.	4.	5.	6.	7.	8.	9.
SiO_2 (com'd)	50.46	50.15	56.42	65.10	39.94	....	40.33	29.67	44.20
Al_2O_3	35.90	35.60	26.35	22.22	36.30	0.72	38.54	20.87	39.14
H_2O	12.74	13.61	10.95	7.10	14.52	0.35	13.00	8.61	14.05
K_2O	....	....	0.48	0.18	0.42	0.14	0.66	1.55	0.25
Na_2O	....	0.07	....	....	....	....	....	....	....
CaO	0.13	0.11	0.60	0.14	0.19	0.22	0.08	....	....
MgO	0.02	0.16	0.55	0.18	0.19	....	0.38	0.30	....
Fe_2O_3	1.50	0.83	1.33	1.92	0.46	0.18	0.90	1.45	0.45
SiO_2 (free)	....	....	....	....	4.90	98.31	5.15	36.41	0.20
Moisture	....	....	2.80	2.18	3.26	....	....	....	0.90
TiO_2	....	....	1.15	....	....	....	....	1.14	1.05
SO_3	....	0.14	....	....	....	....	....	....	....
Org. matter	....	....	....	0.58	....	....	....	....	....
Total	100.75	100.67	100.63	99.60	99.18	99.92	99.24	100.00	100.24

No. 1.—Mt. Savage fire clay, Md.
No. 2.—Fire clay, Clearfield Co., Pa.
No. 3.—Glenboig clay, England.
No. 4.—Stourbridge clay, England.
No. 5.—Saaran clay, Germany.
No. 6.—"Dinas,"[1] England.
No. 7.—Zettlitz clay, Bohemia.
No. 8.—Stoneware clay, N. J.
No. 9.—Paper clay, N. J.

Building stone, such as granite, limestone, sandstone, slate, brick, etc., are generally subjected to certain mechanical or physical tests in addition to a chemical analysis to determine their relative value.

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[1] Used in making the celebrated "Dinas" Fire Bricks, noted for their endurance at high heats and for swelling and making tight roofs for furnaces.

These physical tests generally comprise :

1. Crushing strength.
2. Absorptive power.
3. Resistance to the expansion of frost, by saturating the stone with water and freezing a number of times to produce an effect similar to frost.
4. Microscopical examination.

1. Crushing Strength.

The crushing strength is generally determined by applying a measured force to one-inch or two-inch cubes of the material until they are crushed.

These compression tests are comparative only and give no idea of the crushing strength of the material in large masses. A Riehlé U. S. Standard Automatic and Autographic Testing machine is used for this purpose.[2] (Fig. 90).

CRUSHING STRENGTH OF VARIOUS BUILDING STONES.

	Ultimate crushing strength.			
	Pounds per square inch.		Tons per square foot.	
Kinds of stone.	Minimum	Maximum.	Minimum.	Maximum.
Granite	12000	21000	860	1510
Trap rock of N. J	20000	24000	1440	1730
Marble	8000	20000	580	1440
Limestone	7000	20000	500	1440
Sandstone	5000	15000	360	1080
Common red brick	2000	3000	144	216

2. Absorptive Power.

This is determined by drying the sample and weighing it, then soaking it in water for twenty-four hours and weighing again. The increase of weight represents the amount of water absorbed. A close fine-grained stone absorbs less water than a coarse-grained one, and generally the less the absorption the better the stone.

ABSORPTIVE POWER OF STONE, BRICK AND MORTAR.

	Ratio of absorption.[1]	
Kind of material.	Maximum.	Minimum.
Granite	1—150	0
Marble	1—150	0
Limestone	1—20	1—500
Sandstone	1—15	1—240
Brick	1—5	1—50
Mortar	1—2	1—10

---

[1] Thus, if 150 units of dry granite weigh after immersion in water 151 units, the absorption is one in 150 and stated 1—150.

[2] For description of this apparatus consult *The Digest of Physical Tests and Laboratory Practice*, Vol. 1, p. 248 (July 1896).

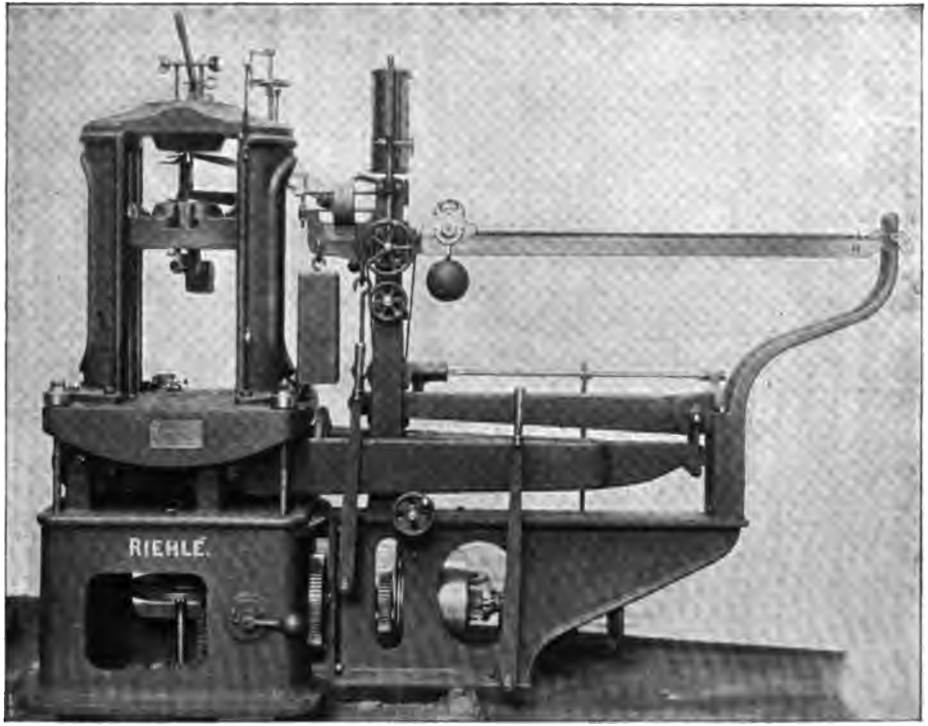

Fig. 90.

3. Freezing Test.

Samples of the weighed material, preferably cut in two-inch cubes, are saturated with water, then placed in a Tagliabue freezing apparatus (Fig. 91) and maintained at a temperature of 10° F. for four hours. They are then removed, allowed to thaw gradually to a temperature of about 65°; then moistened with water and placed again in the freezing apparatus and maintained at a temperature of 10° F. for four hours. This process is repeated at least ten times, when, after the samples have acquired the temperature of the room, the moisture is wiped from them, then dried, and their weight carefully determined. The loss of weight represents the material broken off by the expansive action of freezing the contained water. The following method of making the frost test of building stones, is from "Uniform Methods of Procedure in Testing Building and Structural Materials." by J.

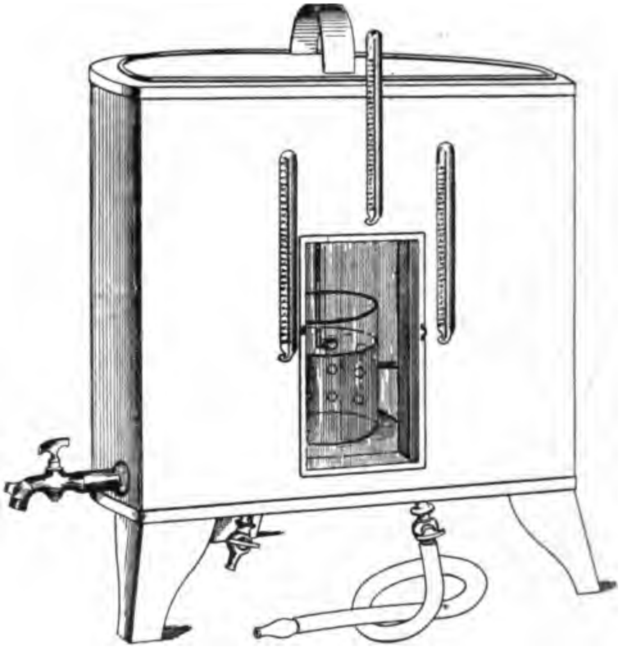

Fig. 91.

Bauschinger (Mechanisch-technischen Laboratorium, München).[1]

The examination of resistance to frost is to be determined from samples of uniform size, inasmuch as the absorption of water and action of frost are directly proportional to the surface exposed. The test sample should be a cube of seven cm. (2.76 inches) length on edges.

The frost test consists of :

a. The determination of the *compressive strength* of *saturated* stones, and its comparison with that of dried pieces.

b. The determination of *compressive strength* of the dried stone after having been frozen and thawed out twenty-five times, and its comparison with that of dried pieces not so treated.

c. The determination of the loss of weight of the stone after the twenty-fifth frost and thaw : special attention must be had to the loss of those particles which are detached by the *mechanical* action, and also those lost by solution in a definite quantity of water.

d. The examination of the frozen stone by use of a magnifying glass, to determine particularly whether fissures or scaling occurred.

For the frost test are to be used :

Six pieces for compression tests in dry condition, three normal and parallel to the bed of the stone, six test pieces in saturated condition, not frozen however ; three tested normal to, and three parallel to, bed of stone.

Six test pieces for tests when frozen, three of which are to be tested normal to, and three parallel to, bed of stone.

When making the freezing test the following details are to be observed :

a. During the absorption of water, the cubes are at first to be immersed by two cm. (0.77 inch) deep, and are to be lowered little by little until finally submerged.

b. For immersion distilled water is to be used at a temperature of from 15° C. to 20° C.

c. The saturated blocks are to be subjected to temperatures of from —10° to —15° C.

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[1] Standard Tests and methods of Testing Materials : *Trans. Amer. Society Mech. Engineers*, 14, 1294.

d. The blocks are to be subjected to the influence of such cold for four hours, and they are to be thus treated when completely saturated.

e. The blocks are to be thawed out in a *given quantity* of distilled water at from 15° C. to 20° C.

The Testing of Brick.—1. When testing bricks as found in a delivery, the least burnt are always to be selected for investigation.

2. Bricks are to be tested for *resistance to compression* in the shape of cubical pieces, formed by the superposition of two half bricks, which are to be united by a thin layer of mortar consisting of pure Portland cement, and the pressure surfaces are also to be made smooth by covering them with a thin coating of the same material. At least six pieces are to be tested.

3. The specific gravity is to be determined.

4. In order to control the uniformity of the material, the *porosity* of the bricks is to be determined; for this purpose they are to be thoroughly dried and then submerged in water until saturated. Ten pieces are to be thoroughly dried upon an iron plate and weighed; then these bricks are to be immersed in water for twenty-four hours, in such a way that the water-level stands at half the thickness; after this they are to be submerged for another twenty-four hours, then to be dried superficially and again weighed; thus the average quantity of water absorbed is determined. The porosity is always to be calculated by volume, though the per cent. of water absorbed is always to be stated in addition.

5. Resistance against frost is to be determined as follows:

a. Five of the bricks, previously saturated by water, are to be tested by compression.

b. The other five are put into a refrigerator which can produce a temperature of —15° C. at least, and kept therein for four hours; then they are removed and thawed in water of a temperature of 20° C. Particles which might possibly become detached are to remain in the vessels in which the brick is thawed until the end of the operation. This process of freezing is repeated twenty-five times, and the detached particles are dried and compared by weight with the original dry weight of brick. Particular attention, by using a magnifying glass, is to be given to the possible formation of cracks or laminations.

c. After freezing, the bricks are to be tested by compression. For this test they are dried, and the result obtained is to be compared with that of dry brick not frozen.

d. Thus, freezing the bricks does not give a knowledge of the absolute frost-resisting capacity; the value of the investigation is only relative, because by it can only be determined which brick can be most easily destroyed by the action of frost.

6. To test bricks for the *presence* of *soluble salts*, five are selected, and again those which are least burnt, and then such which have not yet been moistened. Of these, again, the interior parts only are used, for which reason the bricks are split in three directions, thus producing eight pieces, of which the corners lying innermost in the brick are knocked off. These are then powdered until all passes through a sieve of 900 meshes per square centimeter (about 5,840 per square inch), from which the dust is again separated by a sieve of 4,900 meshes per square centimeter (about 31,360 per square inch), and the particles remaining on the latter are examined. Twenty-five grams are lixiviated in 250 cubic centimeters of distilled water, boiled for about an hour, however replenishing the quantity evaporated, then filtered and washed.

The quantity of soluble salts present is then determined by boiling down the solution and bringing the residue to a red heat for a few minutes. The quantity of soluble salts present is to be given in per cent. of the original weight of brick.

The salts obtained are to be submitted to a chemical analysis.

7. Determinations of the presence of calcium carbonate, pyrites, mica and similar substances are to be made on the unburned clay, for which purpose unburned bricks are to be furnished. These are soaked in water and the coarse particles are separated by passing the whole material through a sieve having 400 meshes per square centimeter. The sand thus obtained is to be examined by the magnifying glass and with hydrochloric acid to determine its mineralogical composition. When impurities, such as carbonate, pyrites, etc., are found, then pieces of brick, such, for instance, as remained from the determina-

tion of soluble salts, are to be examined in a Papin's digester for their deleterious influence. They are to be so arranged in a Papin's digester that they are not touched by the water directly, but are subjected to the action of the generated steam alone. The pressure of steam shall be one-quarter atmosphere, and the duration of test three hours. Possibly occurring disintegration is to be determined by means of the magnifying glass.

4. Microscopical Examination.

This consists in examining under the microscope their sections of the building stone. Important results are often obtained, especially so if the substances used as matrix are indicated—the presence and amount of injurious substances, such as iron pyrites, mica, etc.

Nearly all reports upon samples of building stones now include the microscopical examination.

The first and most essential test applied to building stone is to determine the structure and character of a stone, to know whether it be a granite, syenite, sandstone, quartzite or something else. Although an expert can usually determine at a glance to which, if any, of these groups a particular stone belongs, it is frequently possible to determine the precise lithological character only by a microscopical examination. Thus, for instance, there is a class of Cambrian rocks commonly called Potsdam sandstone, that are not sandstones at all, but are hard, compact rocks known as quartzites, which have been derived from sandstones by metamorphic action. The essential difference between a sandstone and a quartzite lies in the presence of secondary silica between the quartz granules comprising the latter; the presence of this secondary silica or quartz can be determined for a certainty only by microscopical means. The microscope is not only useful in determining the structure of a stone, but it has an even greater practical value in making it possible to detect the presence of deleterious substances, such as pyrite and marcasite, or other minerals whose chemical composition is affected by atmospheric agencies and thus exert a deleterious effect upon the stone.[1]

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[1] H. Lynwood Garrison, *Trans. Amer. Soc. Civil Eng.*, **33**, 88.

Consult :

Tenth Census U. S., 1880. "Building Stones and Quarry Industry."

Stones for Building and Decoration. By G. P. Merrill, 1891.

Building Stone in New York. By Prof. J. C. Smock, in *Bulletin of the New York State Museum*, 1890.

The Testing of Material of Construction. By W. C. Unwin, pp. 410–440.

A Treatise on Masonry Construction. By I. O. Baker, C.E., 1893.

A complete description of the methods of determining the fusibility of Fire Clays will be found in *Trans. Amer. Inst. Min. Eng.*, 24, pp. 42-67.

XXXVIII.

Alloys.

This subject may be divided into three classes:

1. Alloys composed principally of copper and zinc, or of copper, tin and zinc.
2. Alloys or compositions in which copper, tin, lead or antimony are constituents.
3. Alloys not included in the first two divisions.

Alloys of the first class may comprise brass, bronze, bell metal, gun metal, Muntz's metal, etc. The analysis may be performed as follows (if composed of copper and zinc only): Transfer one gram of the brass to a No. 3 beaker covered with a watch-glass, and add *gradually* twenty-five cc. nitric acid; when solution is complete, remove watch-glass, after washing, allow solution to cool, transfer it to a one-quarter liter flask and add water to the containing mark. Mix thoroughly (the solution being at 15° C.) and transfer fifty cc. of the solution to a No. 3 beaker, dilute sufficiently with water and precipitate the copper electrolytically, as in Scheme VI, page 5. Upon complete precipitation of the copper, the platinum cone and spiral are removed from the solution, washed with water, the washings added to the solution in the beaker. Add a few drops of nitric acid to the solution, boil and precipitate the zinc with a slight excess of sodium carbonate. Boil, filter, wash well with hot water, dry, ignite and weigh as ZnO.

Example: One gram brass turnings taken. Solution 250 cc.

Fifty cc. of solution taken :

Platinum cone + Cu	28.175	grams.
Platinum cone	27.995	"
Cu =	0.160	"

$$\frac{0.160 \times 5 \times 100}{1} = 80 \text{ per cent. Cu.}$$

Porcelain crucible + ZnO	17.655	grams.
Porcelain crucible	17.605	"
ZnO =	0.050	"

$$\frac{0.050 \times 65}{81} = 0.040 \text{ Zn}, \ \frac{0.040 \times 5 \times 100}{1} = 20 \text{ per cent.}$$

Cu	80	per cent.
Zn	20	" "
Total	100	" "

Where tin is also a component, the above method is varied as follows:

Take one gram of the fine turnings and digest with nitric acid as above. Evaporate nearly to dryness, add fifty cc. warm water, filter by decantation into a one-quarter liter flask, washing the precipitate thoroughly with hot water, dry it, ignite and weigh as SnO_2 and calculate to Sn.

The filtrate is made up to 250 cc. (15° C.), thoroughly mixed, and fifty cc. taken for copper and zinc as before.

Porcelain crucible + SnO_2	16.6743	grams.
Porcelain crucible	16.5220	"
SnO_2 =	0.1523	"

Sn = 12 per cent.

Platinum cone + Cu	28.115	grams.
Platinum cone	27.995	"
Cu =	0.120	"

Cu = 60 per cent.

Porcelain crucible + ZnO	17.6750	grams.
Porcelain crucible	17.6052	"
ZnO =	0.0698	"

Zn = 28 per cent.

Resumé:

Sn	12	per	cent.
Cu	60	"	"
Zn	28	"	"
Total	100	"	"

EXAMPLES OF ALLOYS OF THE FIRST CLASS.

	Tin.	Copper.	Zinc.	
Bell metal	22	78	..	parts.
Brass	..	72	28	"
Brass (yellow)	..	60	40	"
Bronze for bearings	16	82	2	"
Speculum metal	33.4	66.6	..	"
Delta metal[1] or "Sterro"	..	60	38.2 (1.8Fe)	"
Muntz metal	..	60	40	"

Alloys of the second class may comprise Babbitt metal, Britannia metal, type metal, solder, white metal, camelia metal, Tobin bronze, ajax metal, car-box metal, manganese bronze, magnolia metal, etc.

Analysis of Babbitt Metal.[2]

Five grams of drillings in an eight-ounce beaker are treated with thirty cc. nitric acid (1.20 sp. gr.) and heated till decomposition is complete and the free acid nearly all evaporated. When about five cc. of the solution remain, add fifteen cc. of water, and then add concentrated sodium hydroxide solution till nearly neutral; fifty cc. of sodium sulphide solution are then added, the mixture well stirred, then boiled gently for half an hour. The solution then contains the tin and antimony. The precipitate, which contains the sulphides of lead and copper, is filtered on a nine cm. Swedish filter, and washed thoroughly with water containing one per cent. of the above sodium sulphide solution. The filtrate is received in a 300 cc. beaker.

Tin and Antimony.—The filtrate is diluted to 200 cc. and boiled. Crystals of oxalic acid, C. P., are cautiously added till the sodium sulphide is all decomposed and a milky separation appears, mixed with a precipitate which is usually at first black. Boil for twenty minutes. Pass hydrogen sulphide for ten minutes. Filter rapidly on a Gooch crucible and wash with hot

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[1] Some varieties of Delta metal contain one to two per cent. of tin.

[2] Method of E. M. Bruce, modified.

water. Dry, and heat crucible and contents in a stream of carbon dioxide to a temperature above 300° C. for one hour. Cool in carbon dioxide, remove crucible and weigh as Sb_2S_3. The Gooch crucible containing the $Sb_2S_3 + S$ may be treated with alcohol, then carbon disulphide, then alcohol (in order to remove the sulphur), dried and weighed, instead of igniting in carbon dioxide. $Sb_2S_3 \times 0.71390 = Sb$.

The filtrate from the Sb_2S_3 is treated with thirty cc. concentrated sulphuric acid and boiled down till all oxalic acid is decomposed and strong fumes of sulphuric acid come off. Cool. Dilute cautiously to 200 cc., mix well and filter quickly. Dilute filtrate to 300 cc., warm slightly and pass hydrogen sulphide. Filter stannous sulphide and wash with hot water. Dry, ignite and weigh as stannic oxide in porcelain crucible. $SnO_2 \times 0.788 = Sn$.

The copper and lead sulphide precipitate is washed off the filter, treated with dilute nitric acid, warmed till decomposed, and the sulphur filtered off. The lead is then separated as sulphate by evaporation with sulphuric acid. The lead sulphate is filtered on a Gooch crucible, washed with water containing five per cent. sulphuric acid, dried and ignited over a Bunsen burner. $PbSO_4 \times 0.68298 = Pb$.

The copper is separated from the filtrate by hydrogen sulphide. The sulphide is decomposed by nitric acid, and the resulting solution titrated or electrolyzed.

Sodium sulphide solution for Babbitt analysis is made up as follows: One pound sodium sulphide crystals are dissolved in two liters of water. Portions of this are from time to time saturated with hydrogen oxide gas and filtered for use.

Separation of Tin and Antimony in Alloys.

Mengin treats the alloy (for instance, anti-friction metal) with nitric acid (1.15), collects the insoluble oxides of tin and antimony, washes, carefully ignites and weighs them. *M.* The mixed oxides are next suspended in hydrochloric acid and water and a ball or plate of pure tin added, whereupon the antimony is reduced to metal and the tin converted into chloride; the reaction is best accelerated by heat, about three hours being

Scheme for Analysis of White Metal, Etc., Containing Sb, Sn, Pb, Cu, Bi, Fe, Al, Zn.

<table>
<tr><td colspan="6">Take one gram of the alloy in fine drillings, transfer to a No. 3 beaker, add 15 cc. HNO_3 (1.15), and evaporate nearly to dryness. Allow to cool, add 50 cc. H_2O, 5 cc. HNO_3, boil, allow precipitate to settle, wash with hot water by decantation four times, then transfer precipitate onto the filter and wash with boiling water until reaction of washings is neutral.</td></tr>
<tr><td colspan="2">Residue.[1]—Dry the residue, transfer to a silver crucible, ignite filter paper over crucible; add 8 times the bulk of the precipitate of pure NaOH, fuse 15 minutes, cool, treat the fused mass with water containing 33 per cent., by volume, of alcohol, transfer to a beaker, add 100 cc. of the alcoholic mixture, and stir frequently for 12 hours. Filter, wash with alcohol (33 per cent.), and finally with strong alcohol.</td><td colspan="4">Filtrate contains the lead, copper, zinc, etc., etc. Add 15 cc. dilute H_2SO_4 and evaporate nearly to dryness on the sand-bath, in a porcelain capsule, cool, add 10 cc. H_2SO_4, dilute, warm, filter and wash well with hot water.</td></tr>
<tr><td rowspan="3">Residue contains the antimony as antimonate of soda. Dissolve the residue with 25 cc. HCl containing 3 grams tartaric acid in solution, transfer to a flask, and dilute to 150 cc. Saturate with H_2S, warm, allow to stand in a warm place half an hour, then pass CO_2 through until no odor of H_2S can be detected. Filter, wash with H_2S water, dry, scrape off the precipitate from the filter paper into a small beaker, ignite the filter paper in a porcelain crucible, and heat with a few drops of HNO_3. Add to the precipitate in the beaker 10 cc. HNO_3 heat until oxidation is complete, and transfer to the crucible. Evaporate to dryness and ignite at red heat to constant weight. The amount of Sb_2O_4 thus obtained is calculated to Sb_2.</td><td rowspan="3">Filtrate contains the tin as sodium stannate; evaporate nearly to dryness, until all the alcohol is expelled. Neutralize with HCl, making the solution faintly acid, dilute to 100 cc. with water, saturate with H_2S, filter and wash with a solution containing 5 per cent. acetic acid, 5 per cent. ammonium acetate, till free from chlorides. Dry, scrape precipitate from filter paper into a watch glass; ignite filter in weighed porcelain crucible, oxidize ash with a drop of HNO_3, and ignite; add the precipitate, heat gently at first until no more SO_2 comes off, then heat strongly over the blast-lamp to constant weight. The weight of SnO_2 thus obtained is calculated to Sn.</td><td rowspan="3">Residue, $PbSO_4$.—Dry, ignite in weighed porcelain crucible, observing the direction for this ignition as given in scheme VII, page 9.</td><td colspan="3">Filtrate.—Saturate the filtrate with H_2S, to precipitate the copper: filter and wash with H_2S water.</td></tr>
<tr><td rowspan="2">Residue. CuS.[3]—This is dissolved in nitric acid, boiled, diluted, filtered if necessary, and the copper precipitated by electrolysis, as directed in scheme V, page 7.</td><td colspan="2">Filtrate may contain iron, aluminum, zinc, or (manganese). The solution is boiled to expel H_2S, add a few drops of HNO_3, boil, make alkaline with ammonia, warm and filter, wash well.</td></tr>
<tr><td>Residue, Fe_2O_3. Al_2O_3.—Dry, ignite, and weigh as such. Al can be separated by scheme XIII, page 29. Calculate to Fe and Al.</td><td>Solution may contain zinc; pass H_2S gas to saturation: filter, dissolve the precipitate with HCl, dilute with water, boil to expel H_2S, make alkaline with Na_2CO_3, boil, filter, dry, ignite, and weigh as ZnO, and calculate to Zn.</td></tr>
<tr><td></td><td>Sn.</td><td>Pb.</td><td>Cu.</td><td>Fe. Al.</td><td>Zn.</td></tr>
</table>

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[1] Mengin (*Compt. rend.*, 1894, p. 224) treats the oxides of tin and antimony, as follows: See page 313.

[2] Volumetric determination of antimony in presence of tin. The finely divided alloy is dissolved in strong HCl by heat, adding frequently small quantities of potassium chlorate. The liquid is boiled to remove free chlorine, cooled, a slight excess of strong solution of potassium iodide added, and the liberated iodine separated by standard thiosulphate. 120 Sb liberated 253 iodine, and the weight of iodine found multiplied by 0.475 = weight of Sb. If iron or other metal capable of liberating iodine be present, treat the alloy with nitric acid and evaporate to obtain the oxides of antimony and tin—wash, boil in hydrochloric acid, and proceed as before described. The rationale is, that antimonic chloride is reduced to antimonous chloride, while stannic chloride is not affected. (Sutton's Vol. Anal., p. 134.)

[3] If bismuth be present, it will be precipitated with the copper as sulphide. The nitric acid solution is made alkaline with ammonia, the Bi_2O_3 filtered off and weighed, and the copper determined in the filtrate after acidifying with HNO_3.

necessary for two grams of the oxides. The precipitated antimony is washed by decantation with water, then with alcohol, dried and weighed *A*. There is no appreciable oxidation of the antimony and the method is very exact. The tin is estimated by difference.

$M - A \times 1.262 =$ weight of tin oxide; the latter multiplied by 0.7888 gives the weight of tin in the alloy. An alternative method for the estimation of the tin is to precipitate the latter by zinc. The following figures (indicating grams) of an analysis, show the accuracy of the method:

Samples taken.		Oxides found.	Metals found.	
Sn	1.162	3.131	Sn	1.154
Sb	1.312		Sb	1.309

EXAMPLES OF ALLOYS OF THE SECOND CLASS.

	Iron.	Tin.	Antimony.	Lead.	Copper.	Zinc.	Bismuth.	Phos.
Babbitt metal	....	45.5	13.00	40	1.5	....	....	....
Britannia metal	....	90.0	10.00	....	....	....	....	....
Pewter	....	89.3	7.1	....	1.8	....	1.8	....
Argentine	....	85.5	14.5	....	....	....	....	....
Ashbury metal	....	77.8	19.4	....	....	2.8	....	....
Soft solder	....	50.0	....	50.0	....	....	....	....
Anti-friction metal	....	40.0	5.0	55.0	....	....	....	....
Tobin bronze	0.2	0.9	....	0.4	61.2	37.3	....	....
Phosphor-bronze[1]	....	10.0	....	9.5	79.70	....	....	0.8
Deoxidized bronze	0.20	12.40	....	2.27	82.67	2.45	....	0.005
Magnolia metal	....	4.75	15.0	80.0	trace	....	0.25	....
Rose metal	....	22.90	....	27.10	....	....	50.0	....
Camelia metal	0.55	4.25	....	14.75	70.20	10.20	....	....
Ajax metal	....	10.98	....	7.37	81.28	....	....	0.37
Car-box metal	0.61	....	14.38	84.33	....	0.68	....	....
Parson's white metal	....	86.00	1.00	2.00	2.00	27.00	....	....
"B" alloy, P. R. R.[2]	....	8.00	....	15.00	77.00	....	....	trace

THIRD CLASS COMPRISES:

Aluminum bronze	Al 7.3, Si 6.5, Cu 86.2 or Al 10, Cu 90
Ferro-aluminum	Al 1.23, Fe, etc. 99.73 or Al 12.50, Fe, etc., 87.50
Ferro-tungsten	Fe 43.4, W 53.1, Mn 3.5[3]
German silver	Cu 50, Ni 14.8, Sn 3.1, Zn 31.9
Rosine	Ni 40, Ag 10, Al 30, Sn 20

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[1] Detailed instructions for the determination of phosphorus in phosphor bronze will be found in *The American Engineer and Railroad Journal*, 68, 128.

[2] This alloy, according to C. B. Dudley (*J. Franklin Inst., March, 1892*, p. 168), is the best bearing metal known.

[3] Consult experiments on ferro-tungsten; J. S. DeBenneville, *J Am. Chem. Soc.*, 16, 302.

Metalline	Co 35, Al 25, Cu 30, Fe 10
Aluminum "bourbounz"	Al 85.74, Sn 12.94, Si 1.32
Silicon bronze	Fe, etc. 86.59, Si 13.41[1]
Guthrie's "Entectic"	Cd 14.03, Sn 21.10, Pb 20.55, Bi 50
Arsenic bronze	Cu 79.70, Sn 10, Pb 9.50, As 0.80
Manganese bronze	Cu 88, Sn 10, Mn 2

Aluminum bronze can be analyzed as follows: Take one gram of bronze in fine turnings, transfer to a No. 3 beaker and add gradually twenty-five cc. of aqua regia. Evaporate to dryness, to render the silica insoluble, take up with twenty-five cc. hydrochloric acid, twenty-five cc. water, warm and filter. Wash well. The residue is dried, ignited and weighed as SiO_2, and calculated to Si. The filtrate from the silica is diluted to 250 cc. thoroughly mixed and 100 cc. transferred to a No. 3 beaker and the copper precipitated with hydrogen sulphide, filtered, washed with hydrogen sulphide water, the cupric sulphide dissolved in nitric acid, and the copper determined by electrolysis (Scheme VI). The filtrate from the cupric sulphide is boiled to expel hydrogen sulphide, a few drops of nitric acid added, the solution made alkaline with ammonia, and the alumina determined as in Scheme III, and calculated to Al.

Determination of Manganese in Manganese Bronze.[2] Dissolve five grams of drilling in nitric acid of 1.20 sp. gr., using a large beaker to avoid frothing over. An excess of acid must be avoided as it interferes with the precipitation of the copper by hydrogen sulphide. When solution is complete, transfer to a 500 cc. cylinder without filtering out the precipitated stannic oxide. Make up to 300 cc. and pass a rapid current of hydrogen sulphide from a Kipp's apparatus until the supernatant liquid is colorless. Decant off through a dry filter, 180 cc. corresponding to three grams of sample, and boil rapidly down to about ten cc. Transfer to a small beaker and add twenty-five cc. of strong nitric acid. Boil down one-half, make up with strong nitric acid, boil, and add one spoonful of potassium chlorate. Boil ten minutes and add another spoonful of potassium chlorate. Boil till free from chlorine, cool in water, and filter on asbestos,

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[1] Consult Determination of Silicon in Ferro-silicons; its Occurrence in Graphitoidal Silicon, by H. J. Williams, *Trans. Amer, Inst. Min. Eng.*, 17, 542.

[2] Jesse Jones, *J. Am. Chem. Soc.*, 15, 414.

using filter pump. Wash with strong nitric acid through which a stream of air has been passed. When free from iron, wash with cold water until no acid remains. Place the felt and precipitate in the same beaker and dissolve in ferrous sulphate, using five cc. at a time. Titrate back with permanganate until a pink color remains. Deduct the number of cc. used in titrating back, from the number of equivalents of ferrous sulphate used, and the remainder shows the manganese in the amount of sample taken.

Permanganate Solution.—Dissolve 1.149 grams of potassium permanganate in 1,000 cc. water: one cc. equals 0.001 gram manganese; check by dissolving 0.1425 gram ammonio-ferrous sulphate in a little water and acidulating with hydrochloric acid. This should precipitate ten milligrams of manganese. If not, apply factor of correction.

Ferrous Sulphate Solution.—A solution of ferrous sulphate in two per cent. sulphuric acid so dilute that five cc. corresponds to ten cc. permanganate solution. This is best made by trial and solution.

Analysis of Ferro-Aluminum.

Five grams of the ferro-aluminum are transferred to a 500 cc. beaker and dissolved in seventy-five cc. sulphuric acid (sp. gr. 1.30), then evaporated to dryness. The residue is treated with fifty cc. dilute sulphuric acid diluted to 300 cc. and mixed well. 100 cc. of the solution (= 1.666 grams) are filtered off into a graduated 100 cc. measure; this is then poured into a 250 cc. beaker; about five grams of pure iron wire are now added and the solution boiled, so as to reduce any ferric salt formed; the excess of acid is carefully neutralized with solution of sodium carbonate and the mixture gradually poured into 150 cc. of a boiling solution containing thirty grams of potassium hydroxide and sixty grams of potassium cyanide; the mixture of potassium hydroxide and potassium cyanide with iron precipitated as hydroxide is diluted up to 500 cc. in a graduated measure, and 300 cc. (= 1 gram of sample) filtered off into a six-inch evaporating dish; 200 cc. of a standard solution of ammonium nitrate are now added and the mixture heated forty minutes;

filter and wash the precipitated alumina with hot water, redissolve in twenty-five cc. of dilute hydrochloric acid, dilute to 200 cc., neutralize with ammonium hydroxide, add a slight excess, boil, filter and wash with hot water, dry, ignite and weigh as Al_2O_3. The weight obtained multiplied by 0.534 × 100 = percentages of Al. This amount subtracted from 100 per cent. gives the percentage of iron. (Phillips.)

German silver, Rosine, Aluminum "bourbounz." Guthrie's "entectic" and arsenic bronze all require solution innitric acid to render the tin insoluble, which is then separated by filtration from the other components.

The determination of phosphorus in phosphor-tin presents some difficulty on account of the insoluble compound which phosphoric acid forms with stannic oxide. Hempel,[1] states as follows:

The ordinary way of analyzing phosphide of tin by dissolving it in aqua regia and precipitating with hydrogen sulphide is not satisfactory, as a considerable quantity of phosphorus is thrown down with the precipitated sulphide as a basic phosphate of tin. It is easily analyzed according to Wöhler's method, by treating with chlorine, the chlorides of tin and phosphorus formed being collected in about ten cc. of concentrated nitric acid. If the apparatus be rinsed with a solution of one part concentrated nitric acid and two parts of water, no trace of stannic oxide is precipitated. The phosphoric acid is now easily precipitated in the usual way by molybdic acid.

If dilute nitric acid is taken, a part of the phosphorus separates with the stannic oxide and the result will be too low. This also applies to the determination of phosphorus in phosphor-bronze.

Qualitative Tests of Alloys of Lead, Copper, Tin and Antimony.[2] —For lead, dissolve in aqua regia. If much lead be present, it will separate on cooling as chloride; if only a small amount is present it will be detected by the addition of four volumes of ninety-five per cent. alcohol.

For tin, dissolve in hydrochloric acid, concentrated, and before the portion of alloy taken is completely dissolved, pour off

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[1] *Ber. d. Chem. Ges.*, **22**, 2478, *J. Anal. Chem.*, **4**, 83.

[2] Communicated to the author by G. W. Thompson, Chemist National Lead Co., N.Y.

the supernatant solution, cool to separate lead as chloride, add four volumes of alcohol, filter and to filtrate add slight excess of bromine to convert stannous to stannic chloride; heat to expel free bromine, dilute and pass hydrogen sulphide gas, when if tin is present it will be obtained as yellow stannic sulphide.

For antimony treat alloy with concentrated hydrochloric acid. Almost all the antimony is left undissolved. Decant, wash the residue with water, after which dissolve in hydrochloric acid with potassium chlorate, boil to expel free chlorine, pass hydrogen sulphide, obtaining a precipitate of Sb_2S_5, if antimony is present. If copper is also present, it will be precipitated as copper sulphide and may obscure the color of the antimonic sulphide; if so, filter and treat the precipitate with potassium hydroxide solution, which will dissolve the antimonic sulphide.

Filter and acidify filtrate, when the pure color of antimonic sulphide will be observed if antimony is present.

For copper, treat the alloy with dilute nitric acid in a porcelain dish and evaporate to dryness; if copper is present, it will show as a green ring where it crystallizes out as nitrate on edge of the residue.

For arsenic, dissolve alloy in hydrochloric acid, with addition of potassium chlorate, in an Erlenmeyer flask, boil to expel chlorine, add some more concentrated hydrochloric acid and two grams of sodium thiosulphate, connect flask with a condenser and distil, following in principle the method first proposed by Fischer. Arsenic, if present, will be found in the distillate by passing through it hydrogen sulphide gas.

Quantitative Analysis of Alloys Containing Copper, Lead, Antimony and Tin[1].

One gram of the finely divided alloy is dissolved by boiling in from seventy to 100 cc. of the following solution, in a covered beaker.

The solution is made by dissolving twenty grams of potassium chloride in 500 cc. of water, adding 400 cc. concentrated hydrochloric acid, mixing, and then adding 100 cc. nitric acid of 1.40 sp. gr. No decomposition between hydrochloric acid and nitric acid takes place in this solution in the cold. If complete solu-

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[1] Method of G. W. Thompson.

tion of the alloy is difficult in the amount of solution taken, more is added as required. Continue boiling until solution is evaporated to about fifty cc. Cool by placing beaker in cold water until the bulk of the lead has crystallized out as chloride and then add slowly, with constant stirring, 100 cc. ninety-five per cent. alcohol. Allow to stand about twenty minutes, filter through a nine cm. filter paper into a No. 4 beaker; wash by decantation three times with mixture (4 to 1) of ninety-five per cent. alcohol and hydrochloric acid, concentrated, and wash filter paper twice with the same mixture.

Wash the lead chloride on the paper into a beaker, and wash filter paper several times with hot water, allowing washings to flow into the beaker with rest of the chloride. Finally wash twice with solution of ammonium acetate, hot, (the ammonium acetate solution is made by taking one volume of ammonia water, 0.900 sp. gr., adding to it one volume of water and then eighty per cent. acetic acid until the reaction is slightly acid to litmus), heat until the lead chloride is dissolved, then add fifteen cc. of a saturated solution of potassium bichromate, and warm until precipitate is of good orange color. Filter on weighed Gooch crucible, wash with water, alcohol and ether, dry at 110° C. and weigh.

Evaporate filtrate from lead chloride by heating on hot plate and finally to dryness on water-bath; add ten cc. solution potassium hydroxide (one gram to five cc.) and after a few minutes twenty cc. peroxide of hydrogen; heat on water-bath for twenty minutes, add ten grams ammonium oxalate, ten grams oxalic acid and 200 cc. of water. Heat to boiling, pass hydrogen sulphide with solution near boiling for forty-five minutes; filter at once and wash precipitate with hot water. Boil filtrate to expel hydrogen sulphide, concentrate if necessary and electrolyze over night, using a current of about one-half ampere. Usually by morning the solution will have become alkaline, in which case it may be taken for granted that the tin is all precipitated on the cylinder. The cylinder is removed, washed twice with water and then with ninety-five per cent. alcohol, dried and weighed. The precipitate of antimony and copper sulphides on paper is washed back into beaker with the least amount of water possible, and treated with ten cc. potassium hydroxide solution (1–5), heated on water-bath until undissolved matter is distinctly black; then filtered through the same paper it was washed from into a twelve-ounce Erlenmeyer flask, washed, etc. On the filter the copper is obtained as sulphide with a small amount of lead which failed of precipitation as chloride. If it is desired to determine this lead, it can be done by separation from the copper as usual; if not, dry and ignite precipitate in a small casserole, dissolve in nitric acid, boil to expel nitrogen dioxide, neutralize with sodium carbonate, add a few drops of ammonia, and determine volumetrically with potassium cyanide standardized against pure copper. The solution of antimony sulphide in potassium hydroxide should not amount to over forty cc. Add one gram potassium chlorate, fifty cc. concentrated hydrochloric acid, boil until solution is colorless and free chlorine is driven off; filter through mineral wool; if sulphur has separated into similar flask, wash out original with concentrated hydrochloric acid, cool, add one gram of potassium iodide, one cc. carbon disulphide, and titrate for antimony with tenth-normal sodium thiosulphate, one cc. of which equals 0.0060 gram antimony. This systematic method assumes the absence of other metals than lead, tin, antimony and copper. For the determination of other metals we offer the following suggestions: If arsenic is present it will be separated with the antimony and will liberate iodine, as does antimony. One cc. of tenth-normal thiosulphate equals 0.00375 gram of arsenic. Arsenic is preferably determined on a separate portion by dissolving in hydrochloric acid and potassium chlorate, boiling to expel free chlorine, and distilling after the addition of sodium thiosulphate as a reducing agent, passing hydrogen sulphide through the distillate, and weighing as As_2S_3, or dissolving in potassium hydroxide and determining volumetrically as in the case of antimony.

Bismuth and cadmium sulphides would remain with copper after treatment with potassium hydroxide—this renders this method very suitable for fusible metals. Zinc would interfere with this method, but as zinc does not alloy with lead, we will not speak of it further. Nickel and cobalt alloy but slightly with tin, and if present, should be sought for both in the precipitate left by potassium hydroxide and in the tin precipitated on a cylinder. Iron will also be precipitated with tin if present in an oxalic acid solution. Phosphorus is best determined by Dudley's method.[1]

In alloys containing only lead and tin, with the tin under twenty per cent., the two constituents can best be determined by treatment with dilute nitric acid in a porcelain dish, evaporating to dryness on a water-bath, etc., and determining lead as chromate and tin as stannic oxide. In samples free from iron and copper, antimony may be determined directly by solution in hydrochloric acid and potassium chlorate, boiling to expel chlorine, and titrating as with pure antimony. Antimony in solders may be determined very accurately by dissolving in hydrochloric acid without access of air and filtering out the undissolved antimony on a weighed Gooch crucible. I have not found that a weighable amount of antimony was lost as stibine by this treatment. In the analysis of alloys of lead and tin, Richards' scales,[2] which are accurate within one per cent., may be used. In the examination of the various classes of alloys described at the beginning of this paper, various steps in their analysis may be left out with the absence of the respective metals.

References: "Phosphorus in Phosphor Bronze." By F. Julian., *J. Am. Chem. Soc.*, 15, 115.

"Analysis of American Refined Copper (determination of Cu, Ag, Se, Te, Bi, Sb, As, Fe, Ni, Co, Pb)." *J. Am. Chem. Soc.*, 16, 785.

"The Commercial Valuation of Tin-lead and Lead-antimony Alloys." By J. W. Richards, *J. Am. Chem. Soc.*, 16, 541.

"Materials of Engineering." By R. H. Thurston, Part III.

"The Testing of Materials of Engineering." By W. C. Unwin, p. 342.

"Das mikroskopische Gefüge der Metalle und Legirungen." By H. Behrens, Hamburg, 1895.

XXXIX.

Analysis of Tin Plate.

From two to three grams of the tin plate, cut into strips two to three cm. long by three to five mm. wide, are placed in a dry

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[1] Consult *Am. Engineer and R. R. Journal*, 8, *1894* 128.

[2] Consult *J. Am. Chem. Soc.*, 16, 541.

bulb tube. A current of carefully dried chlorine gas is then passed through the tube, at first in the cold; it is then warmed gently by a Bunsen flame at most three cm. high and placed at least fifteen cm. beneath the bulb, the object being the complete chloridization of the tin, without any attack upon the iron. If the temperature be unduly high, the iron will be violently acted upon and the experiment spoiled. The excess of chlorine, laden with stannic chloride is passed successively through two Peligot tubes and a small Erlenmeyer flask containing water, in which the tin is retained, partly as the tetrachloride, partly as metastannic acid. The connections of these tubes should be entirely of glass and cork, unjointed with rubber and the delivery tube of each part of the apparatus should reach nearly to the bottom, to prevent undue crystallization of the tin salt upon the moist upper walls of the condenser. The current of chlorine must be so regulated that, on the one hand, no stannous chloride is formed, whilst on the other hand, no tin is lost by the chloride being swept through the washing tube; it is continued until the surfaces of the strips are uniformly brown without white spots. Stannic chloride condensing in the narrow portion of the bulb tube is carried forward by the application of gentle heat. The essentials for success are dry chlorine and the minimum temperature possible.—*J. Soc. Chem. Ind.*, *1895*, p. 822.

The Analysis of Tin Plate for Tin, Lead, Iron and Manganese.

The following volumetric method, depending on well known reactions, has given very satisfactory results:

Dissolve five grams of tin or terne plate in 100 cc. hydrochloric acid, 1.10 sp. gr., in a 500 cc. graduated flask, with exclusion of air.

When dissolved, cool and fill up to the mark. Transfer fifty cc. to a beaker, and after adding starch paste titrate the tin with standard iodine solution.

A convenient strength of iodine is made by dissolving 5.38 grams of pure iodine in strong potassium iodide solution and diluting to one liter.

For the iron determination add mercuric chloride in excess to fifty cc. of tin plate solution, and titrate the iron with standard bichromate.

The determination of manganese is quite important, since it shows whether *iron* or *steel* has been tinned.

Treat four grams of tin plate, cut into small pieces, with hot dilute sulphuric acid for about fifteen minutes.

When the iron has dissolved, leaving the layers of tin and lead, add a little zinc and let stand for about two minutes. Filter and dilute to twenty cc.

Take one-half of this filtrate, add five cc. nitric acid of 1.20 sp. gr., and treat in the ordinary way with lead peroxide.

The lead in tin plate is best determined as sulphate after first separating the tin by nitric acid. However, for ordinary work, it is sufficiently accurate to take lead by difference, allowing 0.25 per cent. for phosphorus, carbon, sulphur, silicon, etc., in addition to the tin, iron and manganese previously determined.

In order to test the accuracy of the iodine method for tin, a weighed quantity of pure tin, together with about forty times as much iron, was dissolved and the tin titrated.

The result was as follows:

Amount taken, 0.1255 gram tin.

Amount found, 0.1266 gram tin.

The following are a few analyses that were made of British terne plate used for roofing:

	I.	II.	III.	IV.	V.	VI.	VII.	VIII.	IX.
Tin	1.58	2.08	2.40	3.37	1.60	2.54	1.97	1.96	2.56
Lead......	7.97[1]	7.13[1]	8.89	11.98	2.48[1]	7.48[1]	8.12[1]	7.09	10.23[1]
Iron	89.84	90.23	88.10	84.18	95.31	89.35	89.29	90.55	86.64
Manganese	0.36	0.31	0.31	0.35	0.36	0.38	0.37	0.32	0.32
Carbon..... Phosphorus Sulphur ... Silicon, etc.	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25
	100.00	100.00	99.95	100.13	100.00	100.00	100.00	100.17	100.00

The iodine method may be used for determining tin in all alloys which contain no metals that affect iodine.

However, when the percentage of tin exceeds ten per cent.,

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[1] By difference.

as in the case of solder, the following method, although not quite so simple or rapid, is somewhat more accurate.

In principle the scheme is simply a revision of the well known stannous chloride titration method for iron. Dissolve five grams of the tin alloy in strong hydrochloric acid in a 500 cc. graduated flask, as in the case of tin plate. After diluting to the mark, fill a fifty cc. burette with the solution. Transfer ten cc. of a standard ferric chloride solution (ten grams iron in one liter) to a four-ounce flask and heat to boiling. While boiling run the tin alloy solution cautiously into the ferric chloride until the yellow color disappears. Cool and determine the excess of stannous chloride with standard iodine solution ($Fe_2Cl_6 + SnCl_2 = 2FeCl_2 + SnCl_4$).—*Proceedings Eng. Society of Western Pa.*, **82**, 182.

XL.

Chrome Steel.[1]

The Chrome Steel Company designate its products as follows:

No. 1.—For turning, planing and other tools used for purposes requiring a steady cut.

No. 1 A.—Special for punches, heaters, etc.

No. 3.—For all kinds of fine-edged tools, chipping chisels and machine shop tools; a grade well adapted for general purposes.

No. 2.—Milder than No. 3, for heavy or drop dies of all descriptions, and best quality sledges, etc.

Mill Picks.—Special for mill picks, points, etc.

Rock Drill.—Special for mining, quarry and stone cutting, etc.

Tap and Die Steel.—For tap and dies of all kinds.

Hammer Steel.—Cast Spring Steel.

Machinery Steel.—Of extra toughness and strength, capable of enduring great friction and resisting heavy strains; especially adapted to mandrils, shaftings for rotary pumps, and other purposes where great strength is required.

Round Bars for Prisons or Burglar-Proof Gratings.—These bars consist of alternate layers of steel and iron welded together

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[1] Abstract of Thesis, B. F. Hart, Jr., and J. Calisch: *Stevens Indicator*, 9, 49-65.

and designed for prisons, bank buildings, etc. The gratings or bars are first fitted and then hardened, the steel receiving a temper that will resist any saw, file, or drill; while the iron remaining soft and ductile, will not fracture under heavy blows. This combination of iron and steel is also made in special shapes, and is largely used in safe building. Chrome steel is also extensively employed in the construction of large bridges. Chrome steel possesses great strength, as the following table of tests indicates (page 328). Tests are made by Capt. Eads upon samples of chrome steel furnished in the construction of the Illinois and St. Louis Bridge.

Analysis.

Chromium Determination.—Dissolve two grams of the sample in seventy-five cc. of hydrochloric acid (sp. gr. 1.12) in a 500 cc. flask fitted with a rubber cork containing a glass tube and a Bunsen valve (see page 29); heat gently. During the solution carbon dioxide is passed into the flask slowly to prevent oxidation of the iron. When solution is complete, nearly neutralize excess of free acid with sodium carbonate and render slightly alkaline with powdered barium carbonate. Add distilled water nearly to the containing mark, cork the flask tightly and allow to stand for twenty-four hours, with occasional shaking.

All the chromic oxide and a small amount of ferric oxide are precipitated, whilst all the ferrous chloride, manganese chloride, etc., remain in solution. Filter off the precipitate together with the excess of barium carbonate, wash with hot water, transfer filter paper containing the precipitate to a flask and dissolve in hydrochloric acid with heat.

Filter, wash well, and to the clear filtrate add ammonium hydroxide in slight excess and boil.

The chromic oxide and the ferric hydroxide are thereby precipitated while all the barium remains in solution.

Filter, wash well with hot water, dry, ignite, and fuse in a platinum crucible with sodium carbonate and sodium nitrate. Extract the fused mass with hot water, boil and filter off the residual iron oxide.

The filtrate contains all the chromium as the yellow sodium

Mechanical Tests of Chrome Steel.

Name of Makers.	Length in inches.	Diameter in inches.	Distance between collars in inches.	Limit of elasticity in pounds per square inch.	Modulus of elasticity in pounds per square inch.	Breaking strength in pounds per square inch of original section.	Remarks.
American Tool Steel Co ……	11.640	0.550	5.000	71,600	25,600,000	106,500	For lathe tools.
Butcher & Co ………………	10.875	1.005	6.000	45,300	14,550,000	68,000	Cut from a chrome steel ingot.
" " ………………	8.950	0.560	3.250	42,630	14,868,000	64,900	" " "
" " ………………	9.000	1.003	3.000	35,700	50,000,000	100,100	" " "
" " ………………	11.956	1.005	6.000	43,000	15,450,000	75,950	" " "
" " ………………	11.975	1.010	6.000	40,000	15,600,000	83,827	" " "
" " ………………	10.650	1.010	4.187	49,900	15,598,000	66,160	" " "
" " ………………	10.350	1.010	6.025	44,940	16,900,000	64,290	" " "
" " ………………	10.525	0.567	4.437	47,400	14,615,000	77,400	Bolt from a chrome steel stave.
" " ………………	12.050	0.565	4.100	67,400	20,325,000	99,200	" " "
" " ………………	12.100	0.584	5.000	67,700	13,945,000	104,500	" " "
" " ………………	11.907	0.557	5.875	52,230	13,324,000	110,070	" " "
" " ………………	11.995	0.567	5.900	57,377	15,139,000	100,499	" " "
" " ………………	9.225	0.565	3.000	65,000	11,950,000	107,400	" " "
" " ………………	9.216	0.565	3.000	55,700	24,200,000	107,500	" " "
" " ………………	9.190	0.565	3.000	55,700	16,920,000	105,500	" " "
" " ………………	9.205	0.565	3.000	67,700	15,546,000	103,500	" " "
" " ………………	11.750	0.760	4.000	63,800	22,600,000	147,000	Test bolt chrome steel ingot.

chromate. Evaporate this to dryness with hydrochloric acid in slight excess, and treat residue with hot water.

Filter off any insoluble residue (generally silica), add five cc. hydrochloric acid to the filtrate, then sodium sulphite until the yellow color disappears, and heat to boiling. The chromium trioxide is reduced to chromic oxide. To the boiling solution add ammonium hydroxide in slight excess, boil five minutes, filter, wash well, dry, ignite and weigh as chromic oxide, containing 68.62 per cent. chromium.

Manganese Determination.—Dissolve five grams of the steel in 150 cc. of nitric acid (sp. gr. 1.20). Boil until the bulk is about 100 cc. Add a few crystals of potassium chlorate whereby the manganese separates as manganese dioxide, insoluble in strong nitric acid. Boil for a few minutes, add fifty cc. cold concentrated nitric acid, filter on an asbestos filter, wash three times with concentrated nitric acid, and four times with cold water. Place the asbestos filter containing the precipitate into a beaker, add an excess of hydrochloric acid and boil until all the chlorine is driven off. Dilute with water, filter off the asbestos, wash well, add ten cc. acetic acid to the hot filtrate and neutralize with ammonium hydroxide. Boil, allow the basic acetate of iron to settle. Filter into a flask and to the filtrate add a few cubic centimeters of ammonium hydroxide and then, carefully, sufficient bromine to precipitate the oxide of manganese. Cork and allow to stand twelve hours, filter, dry, ignite and weigh as Mn_3O_4 containing 72.08 per cent. of manganese.

Silicon Determination.—Five grams of the steel are dissolved in thirty cc. dilute sulphuric acid (one part sulphuric acid to two parts water). When solution is complete, add strong nitric acid until no more effervescence occurs. Evaporate to dryness, moisten with hydrochloric acid and dissolve in excess of boiling water. Filter off the silica, wash with dilute hydrochloric acid and hot water, dry, ignite and weigh as SiO_2 containing 46.7 per cent. of silicon. This process is used when tungsten is absent.

Determination of Tungsten.—Dissolve five grams of the steel in a three-inch porcelain evaporating dish with twenty cc. hydrochloric acid (strong) and fifty cc. of strong nitric acid, and evaporate to dryness.

The presence of tungsten is at once indicated by the yellow color of the tungstic acid (WO_3), which separates with the silica. Add fifty cc. water, ten cc. hydrochloric acid, warm and filter; wash with water acidulated with hydrochloric acid to prevent any tungstic acid passing through the filter.

The tungstic acid is then dissolved on the filter in hot ammonium hydroxide and is thus separated from the silica. The filtrate is concentrated so as to allow of its being transferred into a weighed platinum crucible, in which it is evaporated to dryness, ignited and weighed as WO_3.

Carbon, *phosphorus* and *sulphur* are determined by the usual methods in steel analysis.

The following analyses, made in the laboratory of the Institute, show the composition of some of the varieties of chrome steel:

No. 1 Steel.

C	1.1077	per cent.
P	0.0354	" "
Cr	0.7563	" "
Si	0.1292	" "
S	0.0065	" "
Mn	0.0219	" "
Fe (difference)	97.9430	" "
	100.0000	" "

No. 3 Steel.

C	0.7253	per cent.
P	0.0186	" "
Cr	0.5127	" "
Si	0.1754	" "
S	0.0052	" "
Mn	0.0103	" "
Fe (difference)	98.5525	" "
	100.0000	" "

"Magnet Steel."

C	0.9571	per cent.
P	0.0522	" "
Cr	0.4940	" "
W	0.6186	" "
Si	0.0550	" "
Mn	0.0167	" "
S	0.0043	" "
Fe (difference)	97.8021	" "
	100.0000	" "

"ROCK DRILL" STEEL.

C	0.8508	per cent.
P	0.0218	" "
Cr	0.5455	" "
Si	0.1246	" "
S	0.0057	" "
Mn	0.0112	" "
Fe (difference)	98.4404	" "
	100.0000	" "

XLI.

The Chemical and Physical Examination of Paper.

This subject may be conveniently divided into eight sections:

First.—Determination of the nature of the fiber.

Second.—Microscopical examination.

Third.—Determination of free acids.

Fourth.—Determination of the nature and amount of the sizing used.

Fifth.—Determination of the amount of ash and its analysis.

Sixth.—Determination of the weight per cubic decimeter.

Seventh.—Determination of the thickness of the paper.

Eighth.—Determination of the absolute breaking strength.

First.—Determination of the Nature of the Fiber.

The introduction, in late years, of the various kinds of wood fibers in the manufacture of paper has rendered the chemical examination of the same exceedingly difficult.

This is more especially so where the wood fiber has been subjected to chemical treatment, as in the "sulphite process" or the "soda process," before being incorporated in the paper.

Nearly all of the chemical reactions for the recognition of the wood fibers in paper produce certain colors with the various resins in the wood when the reagent is added. While the fiber prepared entirely by the "mechanical" process can be indicated without difficulty, even when mixed with cotton and linen in various amounts, the conditions are greatly altered when the wood fiber has been subjected to bleaching and chemical treatment, since the latter removes much of the resinous matters of the wood and increases the difficulty of the qualitative examination.

The chemical reactions of the fibers produced from the various woods used in paper-making, pine, poplar, and spruce, are identical, qualitatively, with the following reagents.

1. Hydrochloric acid and phloroglucin produce a red color with "mechanical" wood pulp.
2. Aniline sulphate produces a yellow color.
3. Naphtylamin and hydrochloric acid produce an orange yellow color.
4. Anthracene hydrochlorate produces a red color.
5. Phenol Hydrochlorate produces a bluish-green color.
6. Concentrated hydrochloric acid produces a violet color.
7. Pyrrol and hydrochloric acid produce a purple red color.
8. Pyrogallic acid and zinc chloride produce a dark violet color.
9. Nitric and sulphuric acids produce a red color.
10. Haematoxylin solution produces a red color.
11. Alcoholic solution of cochineal produces a blue violet.

Where the wood pulp is composed entirely of "mechanical" wood fiber the above reactions are very marked, and by the aid of the microscope, the varieties of wood can be determined.

Wood pulp produced by the "soda" or by the "bi-sulphite" process gives a much weaker reaction with the chemical reagents used for identification, and in many instances where the pulp has been used many times in paper-making will give no color reactions sufficient for recognition. The amount of "mechanical fiber" in a mixture of "chemical fiber," linen fiber, cotton fiber and "mechanical" fiber in a paper can be determined as follows:

The sample of paper is first boiled in water, then in alcohol, and afterwards digested with ether. After drying, a solution of chloride of gold is added.

Linen, cotton, and "chemical" wood fiber have no reducing action upon the solution of gold; but the mechanical wood fiber immediately reduces gold from the solution, this action being due to the ligno-cellulose remaining in the mechanical wood fiber.

100 grams of mechanical wood pulp, under above conditions, will reduce 14,285 grams of gold.[1]

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[1] Handbuch der Technisch-Chem. Untersuchungen (BOLLEY). 6th Auf., page 1007.

If a sample of paper be submitted for examination as to the fibers used in its manufacture, the following preliminary work is requisite : The rosin, sizing, filling, etc., in the manufactured paper must first be removed. Cut the paper into small pieces, place them in a beaker and digest with a solution of caustic soda (one part caustic soda to thirty of water), at a moderate heat for ten minutes. Pour off the liquid, replace with double the amount of distilled water, and warm ten minutes ; pour off this liquid, and repeat once. Now place the paper in a solution composed of one part of hydrochloric acid and fifteen parts of distilled water and digest ten minutes. Wash a number of times with distilled water, until washings are no longer acid ; then dry.

Suppose the sample of paper so treated to be composed of a mixture of "mechanical" chemical wood fiber, linen, and cotton —a mixture to be found in many samples of good quality of writing-paper.

A sample of the dried paper is tested with solution of gold chloride. If no reduction of gold takes place, the indications point to the absence of mechanical wood fiber. This, however, is not absolute, since, if the paper has been made from "cuttings," "old paper stock," etc., etc., the mechanical wood pulp might have been treated quite a number of times by chemicals in the production of the finer quality of paper, and its lignocellulose destroyed or modified in such a way as to nullify the gold test.

Generally speaking, however, the reduction of the gold chloride is indicative of the presence of mechanical wood fiber.[1] [2]

R. Benedikt[3] gives a method for the determination of mechanical wood fiber in paper, dependent upon the methyl numbers of lignin contained in it. This process has been tested by W. Herzberg[4] with the result that preference is given to the use of gold chloride solution.

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[1] "Ueber die quantitative Bestimmung des Holzschliffs im Papier." von Rich. Godeffroy und Max Conlon ; Mittheilungen aus dem R. K. Technologischen Gewerbemuseum in Wien. 1888.

[2] Mitteilungen aus dem Königlichen technischen Versuchsanstalten zu Berlin, 1892, p. 54.

[3] *Chem Ztg.*, 15, 201.

[4] Mitt. König. tech. Versuchs., 1891, 44—50.

If the amount of mechanical wood fiber in a paper amounts to about ten per cent., Gottstein[1] states that the fibers may be counted under the microscope, after the fibers have first been made visible by a treatment with an alcoholic phloroglucinol solution and hydrochloric acid. Fifteen per cent. or more of the mechanical wood fiber in the mixture renders the test valueless. If chemical wood fiber be present in a paper with mechanical wood fiber, no color tests for the former are positive in the presence of the latter, since the mechanical wood pulps possess a greater tinctorial power.

Should mechanical wood fiber be absent, however, a solution of resorcin can be applied to a properly prepared sample of the paper. Chemical wood fiber produces a violet color, whereas cotton and linen are without action.

A solution of phenol also produces a violet color under similar conditions.

Second.—Microscopical Examination.

By careful manipulation of the microscope, the fibers of linen, cotton, and the various woods can be recognized.

The distinction must be noted here, however, that the fibers from paper, no matter what the source, do not have the appearance under the microscope that they possessed before the mechanical and chemical treatment required in the manufacture of paper.

The chemical process in paper-making is very severe upon the various fibers, since they are subjected to beating and cutting in the "beating-machine," to protracted maceration in strong alkali, to digestion in boiling water, to bleaching with chloride of lime, are loaded with various clays, and finally are sized, and often burnished.

This difference between linen fibers before and after treatment is shown in Figs. 92 and 93.

A comparison shows not only a radical change in the form of the fibers, but a difference in the transparency, due to removal of soluble portions of the fiber.

Poplar wood fiber (Fig. 94) made by chemical process, under

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[1] Papier-Zeitung, 1884, 432.

the microscope resembles the fibers of linen more than does any of the wood fibers. It, however, has one distinguishing characteristic, even among the disintegrated pulps, that is, the tangential fragments have among them particles bearing a grate, or screen-like, appearance, as shown in Fig. 95.

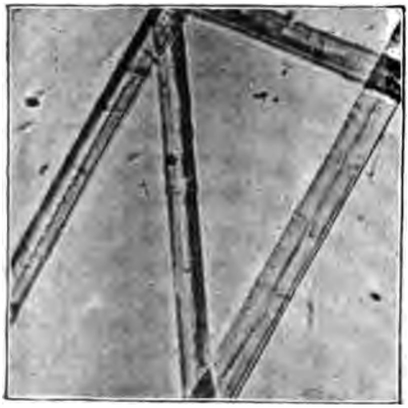

Fig. 92.

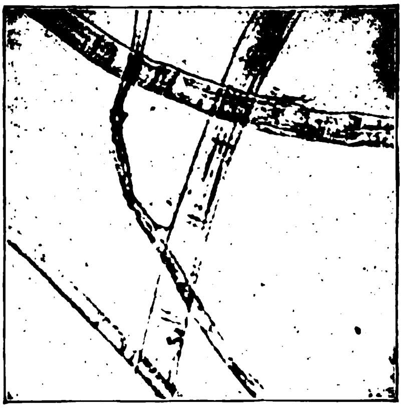

Fig. 93.

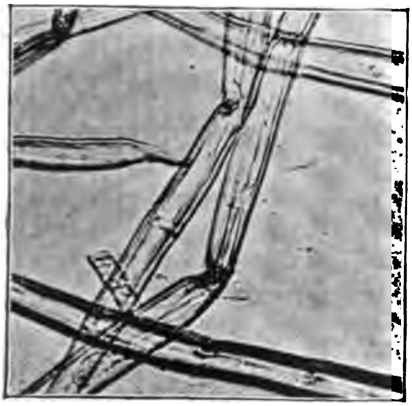

Fig. 94.

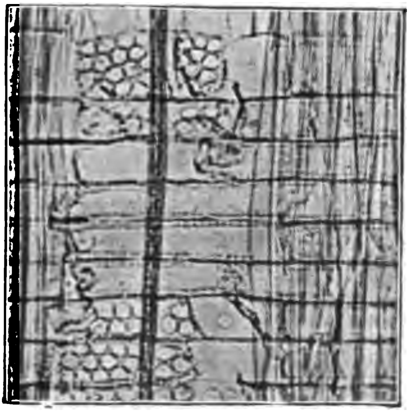

Fig. 95.

The coniferous woods used in paper-making show peculiarities in structure entirely different, under the microscope, from linen and cotton, the most distinctive one being the small circular "pits" or spots along the center of each fiber. A section of spruce wood, composed of fifteen or more fibers, is shown in Fig. 96.

After pulping and making into paper, spruce fiber has the appearance, under the microscope, shown in Fig. 97. It still retains the peculiar circular markings, and is readily distinguishd from the linen paper fiber, Fig. 93, or from cotton fiber, Fig. 98.

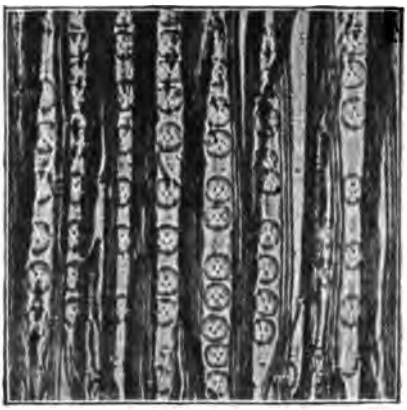

Fig. 96.

Fig. 97.

Fig. 98.

Fig. 99.

In Fig. 99 is shown the peculiar "center-marking" of coniferous fiber, as taken from a sample of writing paper sold as linen paper, but shown by both chemical and microscopical examination to be composed largely of spruce fiber and linen.[1]

The microscope will thus determine the differences between the various fibers used in paper-making, and, by properly arranged apparatus connected therewith, the percentage of each variety of fiber.

According to the German official directions, the sample of paper, after removal of sizing, etc., is to be steeped in a solution of one-fifth gram of iodine and two grams of potassium iodide in twenty cc. of water and then examined under the microscope. The fibers may be conviently divided into three groups :

1. Linen, hemp, and cotton.
2. Wood-cellulose ("chemical" wood fiber), straw-cellulose, and esparto.
3. Ground wood-cellulose and jute.

After treatment with the above solution, the fibers of group 1 are stained brown, those of group 2 are not colored, whilst the strongly lignified fibers of group 3 are colored yellow. But it has been found that this method is somewhat defective; the cellulose of group 2, for example, being invariably to some extent stained, whilst the members of group 1 are so deeply colored that it is almost impossible to distinguish their structural characters. After many experiments, the following method was found more satisfactory.

The paper is placed on the object-glass of the microscope and treated with iodine solution, the unabsorbed iodine removed by means of filter paper, and the paper covered with sulphuric acid dilute. The solution of iodine in potassium iodide should be of such a strength that a layer of three cc. thickness should be of ruby-red color and quite transparent. The paper is now removed and boiled with a solution or dilute potassium hydroxide, washed thoroughly, and replaced on the object-glass. The color reactions are as follows :

1. Cotton, linen, and hemp take a violet-red or wine-red color.

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[1] The micro-photographs used in this article are from specimens made during an investigation upon fibers of papers by Charles S. Shultz and the writer in 1893, and represent the fibers magnified 200 diameters.

2. Well bleached wood-cellulose and ordinary bleached straw-cellulose are colored gray-blue or pure blue, without any tinge of red.

3. Unbleached or imperfectly bleached wood fiber absorbs very little iodine and remains colorless.

4. Strongly lignified fibers, such as ground wood-cellulose and raw jute, are colored yellow.

The numbers of each variety of fiber are now carefully counted by means of the microscope and an eye-piece micrometer ruled in squares. This chemical treatment and microscopical examination is to be repeated upon at least fifty different pieces of paper from different parts of the sample, and an average taken. By this means approximate percentages of each variety of fiber in the paper can be stated.[1]

Third.—Determination of Free Acids in the Paper.

Free acids in the paper may be :

1. Chlorides, from the hypochlorites used in the bleaching, and which have not been removed by the " anti-chlor."

2. Sulphuric acid, from acid alums used in the sizing.

Free acids are exceedingly injurious to the paper, producing gradual deterioration in the breaking strength, and also producing brittleness.

The amount of chlorides can be determined as follows :

Take five-tenths gram of the paper, cut into small portions, and digest with fifty cc. of boiling distilled water for two minutes, then filter. The filtrate is acidified with a few drops of nitric acid, and the amount of chlorine determined by a one-tenth normal silver nitrate solution.

The free sulphuric acid determination requires the determination of the combined sulphuric acid in the alum, since in the titration with soda solution the combined acid, as well as the free, is indicated. The combined acid is determined indirectly and then substracted from the total acid, the difference being the free acid, thus : If the alum used is potash alum, the percentage of potash should be determined, and then the amount of sulphuric acid and alumina calculated from the formula of the alum (anhydrous) $K_2Al_2(SO_4)_4$.

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[1] *J. Soc. Chem. Ind.*, 8, 564.

If soda or ammonia alum be used, the determination of the soda, or ammonia, will be required. Where no clay has been used in the paper, the alum can be determined[1] instead of the other base, and the sulphuric acid necessary to form the alum calculated; this latter is then deducted from the total acid. Total acid is thus determined:

Two grams of the paper are cut into small pieces and digested with 200 cc. of boiling distilled water for three minutes, then filtered and a few drops of solution of litmus added. A solution of tenth-normal soda is gradually added from a burette, until the red color of the solution turns to blue, when the amount of alkali used is noted and calculated to sulphuric acid.

From the total amount of sulphuric acid is subtracted the combined sulphuric acid already determined in two grams of paper. This latter amount is found by determination of any of the bases, alumina, potash, soda, or ammonia, and calculation of the required acid necessary to form the alum used in the paper.

If aluminum sulphate, $Al_2(SO_4)_3$, be used instead of alum, then the free acid and combined acid will be the same in amount, since aluminum sulphate is an acid salt, and titration with the soda solution will give the amount directly.

Fourth.—Determination of the Nature and Amount of Sizing Used.

A paper sized with rosin, when extracted with absolute alcohol, gives a solution which, poured into excess of water, yields a milky turbidity due to precipitated rosin.[2] Another test is based on the Raspail reaction, rosin giving, with sugar solution and sulphuric acid, a violet-red color. The sugar may be omitted, as enough is formed for the reaction by the action of the sulphuric acid on the cellulose of the paper.

The presence of animal size is detected by treating the aqueous extract of the paper with tannin. The following fundamental distinction between papers sized with rosin and gelatin is found to exist. In the former the rosin is distributed uniformly throughout the substance of the paper, while in the latter, whether the sizing has been performed in the pulp or sheet, it is

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[1] Basic sulphate of alumina forms an exception. Ferguson: Basic sulphate of alumina, *J, Am. Chem. Soc.*, 16, 153.

[2] W. Hertzberg: Mitt. König. Tech. Versuchs, 3, 107; *J. Soc. Chem. Ind.*, 9, 99.

always found exclusively on the surface of the finished product. This peculiar property of gelatin can be shown by saturating a plaster-of-Paris slab with gelatin solution colored suitably, and breaking it when dry, on which it will be found to be colored to a trifling depth, the inner part being white. On these facts the following test is based: A half-sheet of paper is repeatedly crumpled and unfolded, and when the surface has been thoroughly chafed, is smoothed out and written upon: if it is sized with rosin, the inscribed characters are but little blurred; while, if animal size has been used, they run freely, and are visible from the opposite side of the sheet. Leonhardi has modified this test, removing the doubtful element introduced by the manual use of pen and ink. A pipette, of which the exit is ten cm. above the paper, and which delivers drops weighing 0.03 gram each, is filled with a solution of ferric chloride containing 1.531 per cent. of iron. A single drop is allowed to fall and to remain on the paper for the same number of seconds that one sq. m. of the paper weighs in grams, when it is removed by blotting paper, and the under side of the paper brought in contact with a plug of wadding wet with a weak solution of tannin: the production of a black color proves the iron solution to have penetrated, and, therefore, shows the sizing to be of animal origin.

Schuman's method for the determination of rosin in paper is as follows: Two grams of the paper are cut into fine pieces and digested below boiling fifteen minutes with a five per cent. solution of sodium hydroxide, and filtered.

The filtrate is made acid with dilute sulphuric acid, the rosin separating and rising to the surface of the liquid. This latter is filtered upon a weighed filter, dried at 100° C. to constant weight, and its weight carefully determined.

Starch was used, formerly, as a sizing for paper, but in recent years it has been largely replaced by rosin size. It can be detected as follows:

The paper is cut into small portions and is digested with boiling water for fifteen minutes, then filtered. To the filtrate is added a drop of a dilute solution of iodine. A blue coloration is indicative of the presence of starch.

The quantitative determination is dependent upon the conversion of starch into glucose by means of dilute sulphuric acid, and estimation by means of Fehling's solution.

Ten to fifteen grams of the paper are digested with 250 cc. of distilled water, to which has been added two per cent. of sulphuric acid. Two or three hours' heating at 100° C. is sufficient to convert the starch into glucose, the exact point being determined by taking a drop of the solution and adding thereto one drop of the dilute iodine; if no blue color is shown, the conversion is complete.

The solution is now made alkaline with soda, diluted with water to 500 cc., and two samples each of 150 cc. taken, filtered, washed well and treated with Fehling's solution,[1] as usual in the determination of sugars. Sadtler states as follows regarding this test:

"In carrying out the gravimetric method the Fehling's solution remains in excess (indicated by the blue color of the solution after boiling), while the cuprous oxide is carefully filtered off and further treated."

The procedure is as follows:[2]

"Sixty cubic centimeters of the mixed Fehling's solution and thirty cubic centimeters of water are boiled in a beaker, and the solution containing the maltose added thereto and the mixture again boiled. It is then filtered with the aid of a filter-pump, upon a Soxhlet filter (asbestos layer in a tared funnel of narrow cylinder shape), quickly washed with hot water, and then with alcohol and ether, and dried. The asbestos filter, with the cuprous oxide, are now heated with a small flame, while a current of hydrogen is passed into the funnel, so that the precipitate is reduced to metallic copper It is allowed to cool in the current of hydrogen, placed for a few minutes over sulphuric acid, and then weighed."

Fifth.—Determination of the Ash.

Three grams of the paper are transferred to a weighed plati-

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[1] Tollen's formula for Fehling's solution is as follows: 34.639 grams crystallized copper sulphate are dissolved in 500 cc. water. 173 grams Rochelle salts and sixty grams sodium hydroxide are dissolved together in 500 cc. of water. Equal volumes of these solutions are mixed when required for use. Ten cc. of this Fehling's solution correspond to 0.0807 gram maltose—or 0.0765 gram starch.

[2] *Sadtler:* Industrial Organic Chemistry, p. 152.

num crucible and ignited until all carbonaceous matter is consumed. The amount of ash is indicative of the use, or not, of mineral filling, such as Carolina kaolin, to increase the weight of the paper. After the correct determination of the amount of the ash, it should be transferred to a 3-inch porcelain capsule, and the scheme on the opposite page used for its analysis.

It is always advisable to test some of the ash, before its analysis, by fusing a portion on charcoal with sodium carbonate. By this means, lead or chromium can be detected, and then properly separated in the analysis of another portion of the ash. If clay, in appreciable quantities, is found, it will be necessary to add ten per cent. of its weight as water, since most clays contain from eight to twelve per cent. of water, which, in the above instance, would have been driven off during the ignition of the paper to determine the per cent. of ash. If much iron be found, Prussian blue, Indian red, Venetian red, or ochre may have been used. If the color of the ash is blue, ultramarine is present; if white, silica, or a fine quality of clay, or calcium sulphate, or agalite[1] may be present; the chemical analysis readily showing the one used as a filler.

If the ash found is very small in amount, it will be necessary to subtract the amount of ash corresponding to the variety of fiber or pulp with which the paper is made, to exactly determine the amount of ash belonging to the added materials.

ASH IN COMMERCIAL PULPS.

Sulphite	0.48 per cent.
Sulphite, bleached	0.42 " "
Soda	1.34 " "
Soda, bleached	1.40 " "
Straw	2.30 " "
Straw, bleached	1.34 " "
Ground wood (pine)	0.43 " "
Ground wood (fir)	0.70 " "
Ground wood (aspen)	0.44 " "
Ground wood (lime)	0.40 " "
Linen	0.76 " "
Linen, bleached	0.94 " "
Cotton	0.41 " ".
Cotton, bleached	0.76 " "

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[1] A variety of talc—silicate of magnesium—in a finely powdered condition; it has a very extensive use as paper filler.

Transfer to a three-inch porcelain capsule, add fifteen cc. HCl, ten cc. HNO_3, and evaporate to dryness. Add ten cc. HCl, fifty cc. H_2O, boil and filter, and make filtrate up to 250 cc. at 15° C., and thoroughly mix.

<table>
<tr>
<td colspan="4">Residue.—Dry, ignite and weigh.
If clay or calcium sulphate be present, fuse with eight times its volume of Na_2CO_3 in platinum dish. Dissolve in 100 cc. H_2O, acidify with HCl, and evaporate to dryness; add 10 cc. HCl, 50 cc. H_2O, boil and filter.</td>
<td colspan="3">Take 100 cc. for determination of soluble bases, Al_2O_3, Fe_2O_3, CaO, MgO, etc. Add ammonum hydroxide and ammonium chloride in slight excess, boil and filter, wash well.</td>
<td rowspan="4">SO_3.
Take 50 cc., bring to a boil, add slight excess of solution of barium chloride, and set aside four hours. Filter, wash well, dry, ignite and weigh as $BaSO_4$, and calculate to $CaSO_4$.</td>
</tr>
<tr>
<td rowspan="3">Residue.
SiO_2. Dry, ignite, and weigh as SiO_2.</td>
<td colspan="3">Filtrate. — Make alkaline with $NH_4OH + NH_4Cl$, boil and filter.</td>
<td rowspan="3">Residue.
Al_2O_3, Fe_2O_3. Dry, ignite, and weigh as Al_2O_3 if the residue is white; if red, as Al_2O_3, Fe_2O_3. To separate Fe_2O_3 from Al_2O_3, use the method of fusion with KOH in a silver dish. The aluminum forms soluble potassium aluminate. the iron remaining as insoluble Fe_2O_3.
Treat with water, filter, wash well, dry, ignite, and weigh as Fe_2O_3, and subtract weight from total of Al_2O_3, Fe_2O_3. The difference is Al_2O_3.</td>
<td colspan="2">Filtrate.—Add solution of ammonium oxalate in slight excess; set aside four hours, filter, wash well.</td>
</tr>
<tr>
<td rowspan="2">Residue.
Al_2O_3. Dry, ignite, and weigh as Al_2O_3.</td>
<td colspan="2">Filtrate.—Add solution of ammonium oxalate, set aside four hours, then filter.</td>
<td rowspan="2">Residue.
CaC_2O_4. Dry, ignite and weigh as CaO.</td>
<td rowspan="2">Filtrate.—If only magnesia is to be determined in this solution, add sol. Na_2HPO_4, set aside two hours, filter, dry, ignite, and weigh as $Mg_2P_2O_7$. If, however, silicate of soda has been found, then proceed to determine the MgO and Na_2O, as follows: Evaporate the filtrate from the CaC_2O_4 to dryness in a platinum dish, with H_2SO_4, ignite to constant weight. This gives the weight of $MgSO_4$ and Na_2SO_4. Dissolve in H_2O, acidify with HCl, make alkaline with ammonium hydroxide, and precipitate the Mg with Na_2HPO_4, filter, dry, ignite, and weigh as $Mg_2P_2O_7$, and calculate to $MgSO_4$. This latter weight subtracted from total $MgSO_4$ and Na_2SO_4, gives the weight of Na_2SO_4. The latter is calculated to Na_2O.</td>
</tr>
<tr>
<td>Residue.
CaC_2O_4; dry, ignite, and weigh as CaO and calculate to $CaSO_4$.</td>
<td>Filtrate.
Add solution Na_2HPO_4; stir well, set aside two hours, then filter, dry, ignite precipitate, and weigh as $Mg_2P_2O_7$. Calculate to $MgSiO_3$.</td>
</tr>
<tr>
<td>SiO_2.</td>
<td>Al_2O_3.</td>
<td>$CaSO_4$.</td>
<td>$MgSiO_3$.</td>
<td>Sol. Al_2O_3. Fe_2O_3.</td>
<td>CaO.</td>
<td>MgO. Na_2O.</td>
<td>$CaSO_4$.</td>
</tr>
</table>

ASH IN FIBERS.

Cotton	0.12	per	cent.
Italian hemp	0.82	"	"
Rhea	5.63	"	"
Best Manilla hemp	1.02	"	"
Sulphite fiber	0.46	"	"
Fine Flemish flax	0.70	"	"
China grass	2.87	"	"
Jute	1.32	"	"
Esparto	3.50–5.04	"	"
Soda fiber	1.00–2.50	"	"

Sixth.—Determination of the Weight per Square Meter.

It is best to use, when possible, five different pieces of the paper (from different packages or rolls), each piece about one square decimeter.

These are placed in a drying oven and exposed to a temperature of 105° C. until the weight becomes constant. The weight of the five pieces, multiplied by 20, gives the weight of one square meter of paper.[1]

Seventh.—Determination of the Thickness.

The thickness of paper can be accurately determined by the apparatus, a sketch of which is shown in Fig. 100.

By means of a delicate spring, a lever, s_2, is held against s_1, touching s_1 only at one point. S_2 carries a toothed segment, which moves a pointer, z, along an arc divided into 500 parts. One division represents 0.002 mm. of thickness of the paper tested.

Eighth.—Determination of Breaking Strength.

By the strength of a paper is understood the measurement of the resistance it offers to breaking or tearing strains. This resistance is always greater in the direction of the length of the web of paper, as it is made on the paper-machine, than across the web. On the other hand, the amount of elongation, which is measured while determining the breaking strain is greater in the direction across the web than parallel to it.[2] The tensile strength of the sheet, both across and parallel to the web, is determined separately, and the average values recorded. To ascertain the

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[1] Leitfaden für Papier-prüfung, W. Herzberg, Berlin, 1888.

[2] Verhandlung des Vereins zur Beförderung des Gewerbefleisses in Preussen, 1885.

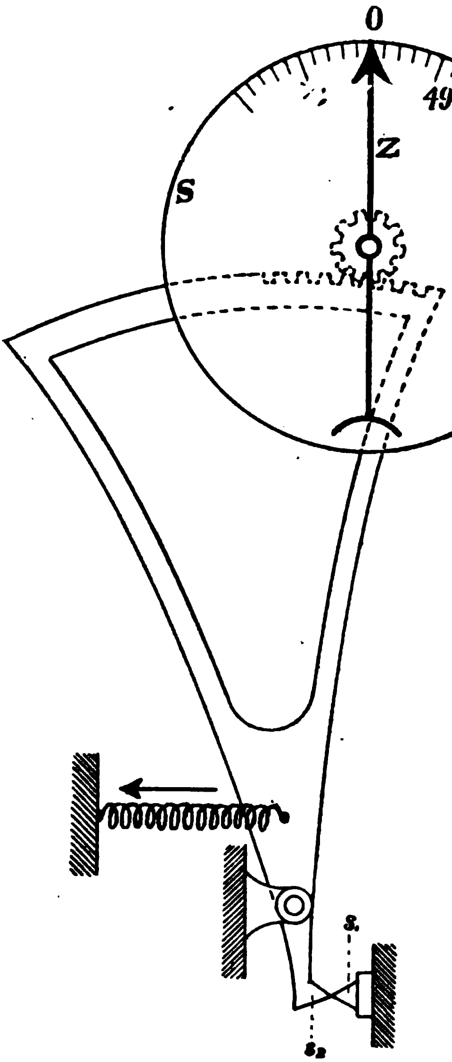

Fig. 100.

direction corresponding to the motion of the paper machine, in any sample of machine-made paper, a circular piece is cut and placed on the surface of water, when it will be observed to roll up. The diameter of the disk where it is not curved indicates the direction of the length of the web. The strips of paper used for ascertaining the tensile strength and elongation are cut to the following size: 180 mm. long by fifteen mm. broad. Five strips, at least, are taken from different sheets and representing the length and across the web, in order to obtain good average values. These strips must be carefully cut; the edges should be smooth and run parallel. Cutting tools are provided for this purpose, consisting of an iron ruler and plates of zinc or glass.

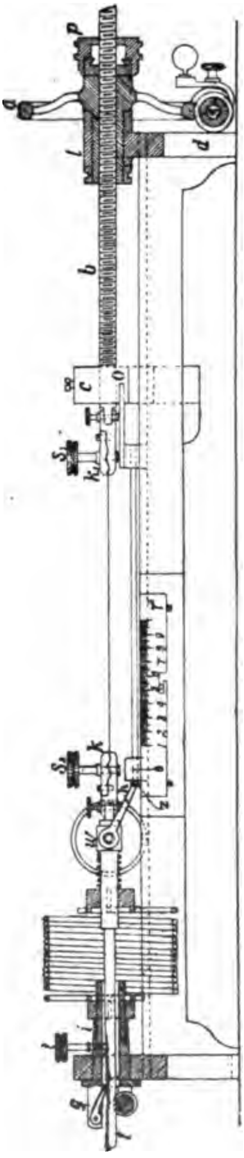

Fig. 101.

Before determining the tensile strength and elongation, careful attention must be paid to the amount of moisture in the atmosphere. The breaking strain of paper decreases with increase of moisture in the air, while under the same influence the percentage amount of elongation increases. The humidity of the atmosphere is very important when testing animal-sized paper and should on no account be overlooked. Indeed, the breaking strain values can only be compared when they are obtained in atmospheres of equal humidity. The percentage of atmospheric humidity chosen is 65, because it is much easier to add moisture to the atmosphere than abstract moisture from it. The former is done by boiling water in the room. The instrument in use for measuring the humidity of the air is the Koppe-Saussure's air hygrometer. Before testing, the strips of paper are placed in the room for at least two hours. The principal machines in use for determining the breaking strength of paper are :

The *Hartig-Rensch*, the *Wendler* and the *Chopper Apparatus*, a description of the *Wendler* being given herewith. This machine is used for ascertaining the strength and elasticity of paper. It consists in the main of four parts. (Fig. 101.)

1. The driver.
2. Apparatus for mounting.
3. Apparatus for transmission of power.
4. Apparatus for measuring force and stretch.

The driving is produced by a hand-wheel, *a*. The hub of this wheel turns in the bearing *l*, which is cast in one piece with the bed *d*. The screw, *b*, is led through this hub, which is hollow, and is fastened to the slide *c*, and through its agency the slide is moved. The hand-wheel is equipped with a bolt-nut, consisting of the shell *p*, and two split nuts, which may be opened or closed by means of a worm, according as the motion of the slide is to be produced by the hand alone or through the agency of the wheel.

The mounting apparatus consists of two clamps kk_1, the first fastened to the carriage *w*, the second to the slide *c*. Between the jaws of these clamps the paper to be tested is stretched, The jaws of these clamps are normal to the axis of stress, wave-shaped, and are lined with leather, in order to prevent the slipping of the strip in the clamps. The jaws are pressed together by means of the screws s_1 s_2.

The transmission of the force is done in this, as in most of this class of machines, by means of a spiral spring, those of Wendler's apparatus possessing respectively a maximum force of nine and twenty kilos. The spring is held at one end by means of the shell *i*, which is fastened to the bed *d*, at the other by the carriage *w*, and passes through the shell *i*. Fastened to the bed by means of screws are the catches *g*, which work in the teeth of the rack, and which, as soon as the paper tears, prevent the spring from flying back.

The measurement of the force is performed as follows:

By means of the lever *h* the carriage pushes the pointer *d* before it, which travels on the graduated bar, *r*. The pointer has a zero mark from which, after the breaking of the paper, the breaking strength is read in terms of kilograms.

The measurement of the elasticity is done by reading the movement of the pointer in the opposite direction along the measuring rod *o*, graduated according to the percentages on a strip 180 mm. in length. After the breaking of the paper, the stretch can be read directly in per cent.

In order to test paper with this apparatus, one adjusts the force measuring rod, by raising the catches, setting the spring in oscillation, allowing it to come to rest and then carefully sliding the pointer down until it touches the lever. Observe whether the zero of the pointer agrees with that of the measuring rod. If this is not the case, the latter is moved until both coincide. The spring is now fastened by means of a screw *t* and the sled is moved until the zero marks of both sled and stretch-measuring rod coincide. Take a piece of the paper to be tested, previously cut to standard size, clamp it in, loosen the screw *t*, drop the catches and begin the experiment, giving the wheel a slow and uniform motion. After breaking the paper, read off the loading as well as the stretch, relieve the spring by holding the carriage still with one hand, loosening the catches with the other and allowing the spring slowly to slide back into place.

In order to insert a new spring, take the carriage and by means of it push the spring in the direction of the screw *t*, turn the spring through 90° and take out the carriage and the rack.

In conducting the experiments, strips 180 mm. long and fifteen mm. broad should be used, and not less than five cut from each direction.

In order to render the result independent of the cross section, use is made of the example of Profs. Reuleaux and Hartig. Using for the measure of strengh of paper the "tearing length," which is the length of a strip of paper of any breadth and thickness, which, if hung up by one end, would break in consequence of its own weight.

Let $x =$ unknown tearing length.
$G =$ wt. of the torn strip (in 0.18 mm. length), in grams.
$K =$ no. of kilos necessary to tear strip.

$$\text{Then } \frac{0.18}{G} = \frac{x}{K}, \quad \text{or} \quad x = \frac{0.18}{G} K.$$[1]

For testing materials which require more power to break than paper, as for instance cardboard, Schopper has constructed a more powerful apparatus, which has a maximum force of 150 kilos. As the apparatus is built on the same fundamental principles as the "Wendler," a description here is needless.

References: "Handbuch der Papierfabrikation." By S. Mierzinski, 1886.

"A Text-Book on Paper-Making." Cross & Bevan, 1888.

"The Art of Paper-Making." Alex Watt, 1890.

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[1] Papier-Zeitung, 1891.

"The Chemistry of Paper-Making." By R. B. Griffin & A. D. Little, 1894.

"Mittheilungen aus den Königlichen technischen Versuchsanstalten zu Berlin," 1891, 1892, 1893.

XLII.

Soap Analysis.

Soaps may be conveniently classified into

Toilet soaps, the finest grades of which contain no impurities or free alkali ;

Laundry soaps, in which rosin and generally an excess of alkali is present either as sodium silicate, sodium carbonate, sodium borate or free alkali ;

Commercial soaps, which may be subdivided into (*a*) soft soaps, potash being the base, and (*b*) "hydrated" soaps, soda being the base, ("marine soap" being an example) formed by caustic soda and palmnut oil or cocoanut oil ; and

Medicated soaps, containing medicinal agents such as carbolic acid, tar, sulphur, etc., etc.

The complete analysis of a soap often presents considerable difficulty—since many adulterants may be used in the cheaper grades, and many substances not adulterants, the use of which is permitted as colorants and for perfume. Allen states that besides the alkali and fatty acids and water requisite for the formation of a soap, the following substances have been found in the different varieties—ochre, ultramarine, sodium aluminate, borax, resin, vermilion, arsenite of copper, alcohol, sugar, vaseline, camphor, gelatin, petroleum, naphthalene and creosote oils, carbolic acid, tar, glycerine in excess, oatmeal, bran, starch, barium sulphate, sulphur, steatite, clay, Fuller's earth, pumice-stone, kieselguhr, chalk, whiting, etc.

The common "yellow soap is formed by the saponification of tallow or palm tree oil with soda, "recovered grease" is also used in the cheaper grades; cotton-seed oil, olive oil, hemp-seed oil, palm oil, cocoanut oil, castor oil, lard, and lard oil, are all used in the manufacture of soap.

The following scheme for soap analysis is by C. R. Alder Wright and C. Thompson.[1]

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[1] *Analyst*, 11, 47.

Scheme for Soap Analysis. (*A.*)

<table>
<tr><td colspan="4">A.—Dry 10 grams of the soap as described on page 352. The loss is water, with possible traces of alcohol. Place the dried soap in a plaited filter and exhaust it with redistilled petroleum ether, in a Soxhlet tube.</td><td colspan="4">H.—Exhaust 10 grams of the sample with 100 to 150 cc. of absolute alcohol, avoiding exposure to air. Methylic alcohol cannot be used.</td></tr>
<tr><td rowspan="3">B.—<i>Solution</i> will contain any unsaponified and unsaponifiable matters, the nature of which can be ascertained by scheme B, page 351. Their total amount may be found by distilling the petroleum ether solution, drying the residue at 100°C, and weighing.</td><td colspan="3">C.—<i>Residue.</i> Allow the adhering petroleum ether to evaporate, and exhaust the residue thoroughly with boiling water.</td><td rowspan="3">I.—<i>Solution.</i> Add a few drops of neutral alcoholic solution of phenolphthalein. If a pink color be produced, titrate cautiously with decinormal acid, the volume of which required corresponds to the free caustic alkali of the soap. If no pink coloration be produced on adding phenolphthalein, titrate the liquid with decinormal caustic alkali, the volume required corresponding to free fatty acid.</td><td colspan="3">J.—<i>Residue.</i> Dry at 100° and weigh. Exhaust thoroughly with boiling water and filter.</td></tr>
<tr><td colspan="2">D.—<i>Solution.</i> Test the hot liquid with a known measure of standard sulphuric acid, using a moderate excess. Agitate thoroughly and pass the separated aqueous liquid through a filter.</td><td rowspan="2">E.—<i>Residue.</i> Consists of insoluble matter, mineral and organic. It may be examined in a similar manner to residue L.</td><td colspan="2">K.—<i>Solution.</i> Divide into two equal parts.</td><td rowspan="2">L.—<i>Residue.</i> Consists of <i>insoluble matter.</i> If considerable, dry at 100°C, weigh, and examine qualitatively.</td></tr>
<tr><td>F.—<i>Solution.</i> Add methyl orange and titrate with standard alkali or sodium carbonates free from chlorides. The difference between the free acid thus found and that previously added gives the equivalent of acid required to neutralize the total alkali of the sample.</td><td>G.—Oily layer consists of fatty and resin acid etc. Consult page —.</td><td>M.—Add methyl orange and titrate with decinormal hydrochloric acid. Volume required corresponds to alkali of carbonated silicate and borate present. Employ neutralized liquid for determining sulphates or to test for starch and gelatin.</td><td>N.—Make special examination for borate, silicate and sulphate.</td></tr>
</table>

Scheme for Soap Analysis. (*B.*)[1]

SYSTEMATIC SEPARATION OF UNSAPONIFIED MATTERS FROM SOAP.

Agitate the solution in petroleum ether with dilute hydrochloric acid and separate.

<table>
<tr>
<td rowspan="3">A.—<i>Acid Solution.</i> Examine for heavy metals. (Pb, Hg, Cu, Zn, etc.,) and alumina which if found, must have existed in the soap as oleates.
Potassium and sodium oleates may also have been dissolved if the soap contained much hydrocarbon. If metals are found at this stage, the amount of fatty acids dissolved by petroleum ether must be corrected to ascertain the fatty acids existing in the soap in a free state.</td>
<td colspan="3">B.—<i>Petroleum Ether Solution.</i> Wash free from mineral acid by repeatedly agitating with small quantities of water. Add some alcohol and titrate liquid with standard alkali and phenolphthalein for estimation of fatty acids. Separate and agitate petroleum ether several times with small quantities of aqueous soda as before.</td>
</tr>
<tr>
<td rowspan="2">C.—<i>Petroleum Ether Solution.</i> Evaporate at a low temperature and observe odor, especially towards the end. Weigh residue and then determine unsaponified fat by Koettstorfer's process. In absence of waxes, the KOH required divided by 0.19 gives the weight of glycerine, which, deducted from whole residue, gives that of the hydrocarbons, wax, alcohols, etc. If desired, these may be isolated and further examined.</td>
<td colspan="2">D.—<i>Alkaline Solution.</i> Evaporate to small bulk, dilute with three measures of strong brine and filter.</td>
</tr>
<tr>
<td>E.—<i>Precipitate</i> consists of sodium salts of fatty acids, existing in the soap either in the free state or as oleates of aluminum and heavy metals.</td>
<td>F.—<i>Solution.</i> Acidulate with dilute sulphuric acid, and separate layer of phenols, or titrate portion of diluted solution with bromine.</td>
</tr>
</table>

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[1] Allen: Commercial Organic Anal., 2, 254.

Water.

For the determination of water, the method of Lowe is employed.[1]

From eight to ten grams of the soap, (which has been reduced to very fine shavings, and represents an average sample), is weighed out between watch-glasses and heated in the air-bath, at first from 60°–70° C., to avoid melting, then at 100°–105° C., to constant weight. In selecting the sample in this, as well as in all subsequent determinations, it is essential that an average specimen be obtained, since the content of water in the different parts of the bar varies considerably.

This is best effected by cutting away about one-third from the end and evenly scraping the cut surface of the remainder until a sufficient amount is obtained for analysis.

If the determination of free alkali is of considerable importance, the soap should be dried in an atmosphere free from carbon dioxide. The loss at 105° C. represents the water together with other volatile constituents, such as alcohol and essential oils, which may be present.

Unsaponified Matter.

For the determination of unsaponified matter,[2] the soap, which has been dried in the manner indicated, is extracted in a Soxhlet extraction apparatus with petroleum ether, which, for this purpose, should boil below 80° C, and should leave no residue upon evaporation. After the extraction is complete, the petroleum ether is distilled off, the residue dried at 100° C and weighed.

In a boiled, well-made laundry soap, there should be no unsaponified matter unless the same has been added subsequently.

In addition to unsaponified fats, foreign matters are sometimes found in the petroleum ether extract, such as a soft paraffin (so-called "Mineral Soap Stock"), waxes, hydrocarbon oils, phenol, etc. If waxes are found to be present, the dried soap should be extracted with boiling toluene, which dissolves the same better than petroleum ether.

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[1] J. F. Schnaible, *J. Anal. chem.*, 4, 147-156.

[2] Allen: Commercial Organic Analysis, Vol. 2.

Total Alkali. Fatty Acids.

The dried soap thus freed from unsaponified matter is next dissolved in hot water, preparatory to determining the total alkali and fatty acids. A pure soap dissolves completely in hot water, and no ordinary product should leave more than a slight residue. If the article examined is a "scouring soap", the insoluble residue will be found to contain quantities of fine sand and sometimes talc. The residue, if appreciable, should be washed by decantation, and eventually brought upon a filter with hot water, dried at 100° C., and weighed, after which, if desired, it can be subjected to further examination.

To the aqueous solution is added an excess of half normal sulphuric acid setting free the fatty acids which rise to the surface. The beaker or vessel in which the precipitation was effected is next cooled with ice-water. When the fatty acids,[1] have solidified, it is best to decant the liquid, remelt with hot water two or three times to remove any enclosed mineral acid, again cool, filter, and wash with cold water until the washings are no longer acid, as shown by litmus.

The filtrate from the insoluble fatty acids contains the total alkali now present as sulphate, the excess of sulphuric acid and any glycerol which may have been present in the soap, if saponification was effected in the cold. The acid liquid may further contain a small amount of soluble fatty acids. It is first titrated with half normal potassium hydroxide using methyl orange as indicator.[2] From the original amount of sulphuric acid added and the number of cc. half normal potassium hydroxide required to neutralize the excess of the same, the total alkali of the sample can be determined.

It is calculated to Na_2O. After the liquid has been rendered neutral to methyl orange (which indicates the mineral acid), phenolphthalein is added and more potassium hydroxide is run in. The number of cc. of potassium hydroxide required for neutralizing to phenolphthalein corresponds to soluble fatty acids and is calculated to caprylic anhydride $\left(\begin{matrix}C_7H_{15}CO\\C_7H_{15}CO\end{matrix}\right)O$, in the absence of

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[1] Bulletin No. 13, Pt. 4, p. 456, U. S. Dept. Agr., Chem. Div.

[2] Allen : Com. Org. Anal., 2, 260.

more definite knowledge as to their nature. The solution is now concentrated and tested for glycerol, which may be determined by evaporating to dryness and extracting with ether-alcohol mixture[1], or else by oxidizing to oxalic acid by means of permanganate[2] (not always applicable).[3]

In soaps containing silicates of the alkalies (a not unusual constituent), the gelatinous silicic acid which separates on the addition of sulphuric acid remains with the fatty acids on filtration. To separate the fatty acids from this as well as other impurities, proceed as follows:

The funnel containing the filter with the fatty acids is placed in a small beaker and heated in an air bath (Allen's method). As the filter dries, the fatty acids pass through it and collect in the beaker below, while all impurities (silicic acid, talc, etc.) remain on the filter. Of course it is necessary to wash the filter, which remains saturated with the fatty acids, with hot redistilled alcohol or petroleum ether, or else exhaust in an extraction apparatus. The alcohol or petroleum ether is distilled off and the residue treated in the same way as the main quantity of fatty acids.

In determining the fatty acids in a soap, it is frequently convenient to extract with ether in a separatory funnel.[4] To do this the soap solution is placed in the funnel and shaken with sulphuric acid and ether. The separated acids are at once dissolved in the ether. The aqueous solution may be drawn off below, the ethereal solution washed with water, the ether evaporated, and the residue dried at 100° C., and weighed.

Since the fatty acids exist in the soap as anhydrides and are weighed as hydrates, it is necessary to multiply the weight found by the factor 0.97, which gives the weight of fatty anhydrides. The fatty acids, after having been weighed, may be titrated with half normal potassium hydroxide, and from these data may be ascertained what portion of the total alkali exists in combination with the acid as soap.

[1] *Chem. Ztg.*, 8, 1667.

[2] *Chem. Ztg.*, 9, 975.

[3] Allen: Com. Org. Anal., 2, 290.

[4] *Chem. News*, 43, 218.

Free Alkali.

To determine the per cent. of free alkali[1] in soap, a separate portion is weighed out and extracted with *neutral* alcohol in an extraction apparatus. The caustic alkali is determined in the alcoholic solution by titrating with half normal hydrochloric acid, using phenolphthalein as indicator. If, however, the soap contains unsaponified fat, as is frequently the case if made by the so-called " cold-process," this method cannot be used, since in alcoholic solution unsaponified fat would be readily saponified by the free caustic alkali present. In such a case the soap must first be dried in an atmosphere free from carbon dioxide at 100° C., the unsaponified matter extracted with petroleum ether, and finally the soap dissolved in alcohol and the free alkali determined in the alcoholic solution as before. The sodium carbonate, sodium silicate, borax, and everything insoluble in alcohol, remains behind in the extraction tube and may be dried at 100° C. and weighed. If considerable, it may be further treated, as follows:

First, it should be exhausted with boiling water; one-half of this solution is then titrated with half normal hydrochloric acid using methyl orange as indicator. The amount of acid required corresponds to carbonate, silicate and borate. In this solution sulphates may also be determined and starch and gelatine tested for. The other half of the solution is examined qualitatively for carbonate, silicate and borate. If there remains a considerable residue insoluble in water, it may be dried at 100° C., weighed and further examined.

Resin.

Resin is a very common constituent of soaps, the resinates of the alkalies having a similar action to soaps, and the cheapness of the material often suggesting a partial substitution of it for the natural fats and oils.

As a qualitative test for resin, Gottlieb's[2] method is reliable and easily made.

The soap is dissolved in water and heated to boiling. A strong solution of magnesium sulphate is added until the fatty acids are

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[1] Allen: Com. Org. Anal., 2, 251.

[2] Benedikt: Analyse der Fette u. Wachsarten, p. 121.

completely precipitated. The magnesium resinates remain in solution. After boiling two or three minutes, the solution is filtered and the hot filtrate acidified with dilute sulphuric acid. In the presence of resin the liquid becomes turbid, due to the separated resin acids. The boiling should be continued for one-half hour, to make sure that the turbidity is due to resin acids and not to volatile fatty acids. One method for the quantitative determination of resin in soap is that of Hübl,[1] as follows:

One-half to one gram of the mixture of fatty and resin acids is heated in a closed flask on the water-bath with about twenty cc. of alcohol to complete solution. The acids are neutralized with alkali, using phenolphthalein as indicator. The alcoholic soap solution is then poured into a beaker, the flask rinsed with water, the solution diluted to 200 cc., and silver nitrate added to complete precipitation. The precipitate (consisting of the silver salts of the resin and fatty acids) must be protected from sunlight. It is filtered, washed with water, dried at 100° C., and extracted in a Soxhlet tube with ether. The silver resinates dissolve in the ether, while the silver salts of the fatty acids remain behind. The ethereal solution, as it leaves the extraction tube, should be yellow or light brown in color, but not dark brown. It is filtered, if necessary, and the filtrate shaken with hydrochloric acid in a separatory funnel. The resulting ethereal solution of the resin acids is filtered from the silver chloride, washed with water, and the filter and separator rinsed with ether, the ether distilled off, and the residue dried at 100° C. As the resin is weighed in the hydrated form, its weight must be multiplied by the factor 0.9732 to obtain the weight of the anhydride.

Twitchell's method for the determination of resin in a mixture with fatty acids depends upon the formation (in alcohol solution) of the ethereal salts of the latter when treated with hydrochloric acid, resin being unacted upon. The gravimetric method is as follows:[2] Two or three grams of the mixture of fatty acids and resin are dissolved in ten times their volume of absolute alcohol and dry hydrogen chloride is passed through in a moderate stream,

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[1] Benedikt, p. 125.

[2] *J. Anal. Appl. Chem.*, 5, 379; Vultè: *School of Mines Quarterly*, 13, 249.

the flask being placed in a vessel with water to keep it cool. The gas is rapidly absorbed, and after about forty-five minutes the ethereal salts separate and float on the solution. After waiting for half an hour longer, the liquid is diluted with five times its bulk of water and boiled until the acid solution is clear, the ethereal salts, with resin in solution, floating on top. To this is added some light petroleum, and the whole transferred to a separatory funnel, the flask being washed out with light petroleum. The acid liquid is then run off, and the petroleum ether solution washed once more with water and then treated in the funnel with a solution of a half gram of potassium hydroxide and five cc. of alcohol in fifty cc. of water. The resin is immediately saponified, and the two layers separate completely. The resin soap solution can then be run off, and the resin recovered, as usual by the addition of an acid. The first stages of the volumetric method are similar to those of the gravimetric, with the exception that the contents of the flask are washed into the separating funnel with ether instead of light petroleum, and the ethereal solution is then thoroughly washed with water until all soluble acidity is removed; fifty cc. of neutral alcohol is then added, and the solution titrated with standard solution of sodium hydroxide.

It is frequently of interest to know the origin of the fatty acids of a soap which is, however, in many cases, a problem not easily solved. The only clues are to be sought in the specific gravity, combining weight, melting and solidifying points, and iodine number of the fatty acids.

The values for the specific gravities in column III page 358, were obtained with a Westphal and a Reimann's balance plummet, with a thermometer of a range 95°—101° C., as shown in accompanying figure.

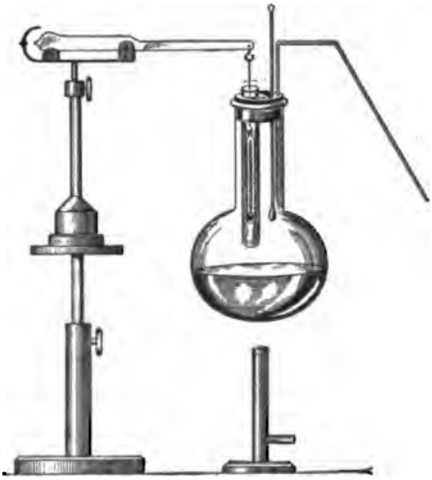

Fig. 102.

Table for the Physical and Chemical Investigations of Fats and Fatty Acids.[1]

Kind of fat.	I.	II.	III.		IV.		V.		VI.		VII.		VIII.		IX.		X.	
	Amount of fatty acids in the fat at 100° C.	Specific gravity of fat at 100° C., determined with König's butter spindle.	Specific gravity at 100° C., determined with Reimann's apparatus in conjunction with the Westphal balance		Refractive index at 60° C.		Polarization at 50°-60° C.		Melting point.		Solidifying point.		Saponification value.		Iodine value.		Volatile fatty acids.	
			Fat.	Fatty acids.	Fat.	Fatty acids.	Fat.	Fatty acids.	Fat.	Fatty acids.	Fat.	Fatty acids.	Fat.	Fatty acids.	Fat.	Fatty acids.	Fat.	Fatty acids.
	Per Cent.								°C.	°C.	°C.	°C.						
Butter	89-91	0.865-0.867	0.899-0.900	0.876-0.888	1.445-1.448	1.337-1.439	0	0	28-33	38-40	20.23	33-35	225-230	210-220	28-32	28-31	22-32	8-13
Cotton-seed oil	96.0	0.8735	0.9038	0.8805	1.457	1.446	0	0	....	35.40	....	32-35	194-195	201.6	106-107	112-115	0.95	0.3
Cocoa butter..	97.0	0.8580	0.8910	0.8780	1.4496	1 4420	0	0	32-33	49-50	23.0	46-47	198-200	190	34	32.6	0.8	0.35
Cocoanut oil..	97.0	0.8700	0.9060	0.8885	1.4410	1.4295	0	0	23-24	24-25	22.23	20.0	255-260	258	9.0-9.5	8.5-9.0	7.5	7.0
Earth-nut oil..	98.0	0.8640	0.8976	0.8830	1.4545	1.4461	0	0	....	30-32	....	29-30	194-196	201.6	94-96	96-97	0.4	0.3
Mutton tallow.	97.0	0.8590	0.8946	0.8673	1.4501	1.4374	0	0	44-55	46-47	40-41	39.0	192-195	210.0	43-44	34.8	1.2	0.4
Cod-liver oil (medicinal).	98.0	0.8715	0.9075	0.8858	1.4621	1.4521	0	0	....	50-52	....	....	175-185	207.0	128-130	130.5	0.4	0.6
Linseed oil....	98.0	......	0.9170	0.8890	1.4660	1.4546	0	0	....	17.0	....	13.5	190-192	196.0	177-178	155.0	0.9	0.4
Almond oil....	98.0	0.8675	0.9015	0.8750	1.4555	1.4461	0	0	....	14.0	....	5.0	190-192	204.0	82-83	87-90	0.55	0.5
Margarin	95-96	0.8590	0.897-0.899	0.876-0.879	1.433-1.453	1.443-1.444	0	0	32-35	42.0	20.22	39.0	192-200	ca.188.0	48-64	......	0.3-0.9	0.7
Poppy-seed oil	96.0	0.8725	0.9075	0.8830	1.4586	1.4506	0	0	....	20.5	....	16.5	193-194	199.0	134-135	116.3	0.6	0.4
Olive oil.......	96.0	0.8640	0.8997	0.8683	1.4548	1.4410	0	0	....	26-28	....	21-22	191-193	193.0	82-83	87-88	1.5	0.25
Palm oil.......	98.0	0.8600	0.8936	0.8790	1.4510	1.4441	0	0	36-37	47-48	....	42-43	201-202	204.0	51.5	53.3	0.6	1.1
Palm-nut oil ..	97.0	0.8670	0.9005	0.8760	1.4431	1.4310	0	0	25-26	20.7	....	....	246-250	264.0	13-14	12.0	3.4-5.0	4.8
Castor oil......	92.0	......	0.9465	0.9290	1.4636	1.4546	+6.4°	+7.8°	....	13.0	....	3.0	201-203	282.0	93-94	87-88	4.0	3.0
Beef tallow....	96-97	0.8600	0.8945	0.8660	1.4510	1.4375	0	0	43-49	45.0	37.0	43-45	193-195	201.6	38-40	26-30	1.0	0.5
Rape-seed oil (crude)	96.0	0.8635	0.8990	0.8715	1.4667	1.4491	0	0	....	20.0	...	16.0	177-179	185.0	98-100	97-99	0.9	0.6
Lard	96-97	0.8610	0.8955	0.8700	1.4539	1.4395	0	0	26-32	37-38	26.0	35.0	195-196	204-207	59-60	47.6	1.1	0.4
Sesamé oil....	96.0	0.8705	0.9043	0.8786	1.4561	1.4461	+1.0°	+1.3°	....	25-32	....	23.5	192-193	201.6	103-105	110-111	1.2	0.4
Sunflower oil..	97.0	......	0.9070	0.8880	1.4611	1.4531	0	0	....	23.0	....	17.0	193-194	201.6	129	133-134	0.5	0.3

[1] W. Thörner: *Chem. Ztg.*, 18, p. 1154-55.

Occasionally, fats, before being used in soap-making, are bleached by various chemical agents, the most common of which are, perhaps, potassium dichromate and hydrochloric acid, or sulphuric acid. If now such a mixture is heated in bleaching, as is frequently the case, the potassium dichromate acting on the hydrochloric acid liberates chlorine, and under favorable conditions, the chlorine combines with the unsaturated acids present in the fats as glycerides, thus utterly destroying the value of the iodine number, the most definite index as to the origin of the fats. Again, it frequently occurs that a mixture of two or more fats may be used, the combining weights, iodine number, and other properties of which closely approximate those of an individual fat, and so an erroneous conclusion may be drawn from an examination of such mixed fatty acids. If, however, a mixture of two fats, in their natural state, without having undergone any bleaching or refining process, is used, it is generally possible to ascertain, with considerable accuracy, the nature of the fatty acids by means of the iodine number, it having been found by actual experiment that the iodine number of a mixture of two fats corresponds within limits of analytical error with the theoretical numbers calculated for the pure fats.

Glycerine in fats and soaps can be determined as follows:[1] three grams are saponified with an alcoholic potash solution, the soap solution diluted to 200 cc., decomposed with dilute acid, filtered from insoluble fatty acids, and the filtrate and washings, which should amount to above 500 cc., evaporated rapidly down to 250 cc., sulphuric acid added and titrated with standard potassium bichromate.

For the titration by bichromate the following solutions are required:

1. Bichromate solution containing about 74.86 grams of potassium bichromate and 150 cc. strong sulphuric acid per liter. The oxidizing value of the solution must be ascertained by titration with solutions containing known amounts of iron wire.

2. Ferrous ammonium sulphate solution containing about 240 grams per liter.

3. A bichromate solution one-tenth as strong as the first.

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[1] O. Hehner: *J. Soc. Chem. Ind.*, **8**, 4.

The ferrous solution is standardized upon the chromate solution, and the glycerol value of the chromate (contents of bichromate divided by 7.486) is calculated. One and five-tenths of the glycerol or soap lye is weighed into a 100 cc. flask, and a little silver oxide added to remove any chlorine or aldehydic compounds. After slight dilution, the sample is allowed to stand with the silver oxide for about ten minutes. Basic lead acetate is then added in slight excess, the bulk of the fluid made up to 100 cc. and a portion is filtered through a dry filter.

Twenty-five cc. of the filtrate are placed in a clean beaker, then forty to fifty cc. of the standard bichromate solution accurately measured, are added, and fifteen cc. strong sulphuric acid. The beaker is covered with a watch glass and heated for two hours in boiling water. The excess of bichromate solution is then titrated back with the ferrous ammonium sulphate solution.

The table of analyses of soaps on the following page comprises in each instance a complete analysis.

In most analyses of soaps the following determinations only are made: Water, alkali combined as soap (Na_2O), alkali free as sodium hydroxide, sodium carbonate, and total fatty acids as anhydrides. Thus, an ordinary yellow laundry soap, analyzed by Schnaible, gave:

Water	19.12	per cent.
Alkali, combined as soap, Na_2O	8.57	" "
Alkali free, as NaOH	0.20	" "
" Na_2CO_3	0.20	" "
Insoluble in H_2O	0.20	" "
Fatty anhydrides	52.32	" "
Resin	19.45	" "
Total	100.00	" "

Washing Powders.

The washing or soap powders contain besides powdered soap, a large percentage of sodium carbonate, usually in the form of dried soda crystals. These powders are generally prepared as follows: Anhydrous sodium carbonate or anhydrous soda ash is added to a "clear boiled" soap paste, and after thoroughly

Analyses of Soaps.

Description of soap.	Origin.	Fatty and resin anhydrides.	Soda (Na_2O) existing as soap.	Silica.	Soda as silicate.	Sodium carbonate and hydrate.	Sodium chloride.	Sodium sulphate.	Lime and iron oxide.	Water.	Total.	Fatty and resin acids.
White No. 1	Tallow.	69.06	8.98	0.01	none	0.27	0.49	0.16	0.07	21.14	100.18	71.20
White No. 2	Tallow and cocoa-nut oil.	60.50	6.82	0.06	none	0.06	0.11	0.12	0.16	32.30	100.03	62.36
Cold water	Tallow, resin and cotton-seed oil.	71.30	7.98	1.07	0.48	0.75	0.36	0.30	0.16	17.44	99.84	73.50
Palm oil	Palm oil.	59.28	6.65	0.42	0.01	0.39	0.47	0.13	0.16	32.35	99.86	61.08
Olive oil No. 1	Olive oil.	71.20	7.58	0.06	0.03	0.22	0.66	0.17	0.20	19.70	99.82	73.40
Pale resin No. 1	Tallow and resin.	60.90	7.22	0.04	none	0.10	0.46	0.12	0.02	31.22	100.08	62.78
Pale resin No. 3	Tallow and resin.	39.92	4.70	0.62	0.25	0.20	1.48	0.18	0.15	52.40	99.90	41.15
Marine	Palm-nut oil.	19.42	3.11	9.00	3.98	3.00	5.13	0.35	0.16	53.32	97.47	20.02
White castile	....	76.7	9.14	...	...	0.09	0.36	...	0.09	13.25	100.54	....

mixing, the somewhat stiff material is drawn off into cooling frames.[1] The cold and hard soap thus formed is then finely ground.[2]

The composition varies greatly. Only a small proportion of resin soap can be used, as such a soap is sticky and cannot be powdered.

Olein soap is generally used and is saponified with sodium carbonate.

References.—"Die Darstellung der Seifen, Parfumerien und Cosmetica." By C. Deite, 1867.

"The Art of Soap-Making." By A. Watt, 1887.

"The Manufacture of Soap and Candles." By W. T. Brandt, 1888.

"Lard and Lard Adulteration." By H. W. Wiley, 1889.

"Die Untersuchungen der Fette, Oele and Wachsarten." By C. Schaedler, 1890.

"Analysis of Washing Powders." *Am. Chem. J.*, 14, 623.

"Soap Powders." *Seifen, Oel und Fett Industrie*, 3, 973.

"Oils, Fats, Waxes and their Manufactured Products." By Alder Wright, F.R.S., 1894.

"Oils, Fats, and Waxes." By Dr. R. Benedikt and Dr. J. Lewkowitsch, F.C.S., 1895.

XLIII.

Technical Examination of Petroleum.

This is usually performed by fractional distillation of the petroleum into three classes of distillates.

1. Light oils, distilling over up to 150° C.
2. Illuminating oils distilling over from 150° C. to 300° C.
3. Residuum.

The method of Engler, which is largely used for this purpose, requires a glass flask of the form shown in Fig. 103. The measurements given in the figure are stated in centimeters. The flask is connected with a condenser in the usual manner.

100 cc. of the oil are taken and the temperature in the flask so regulated that two and one-half cc. of the distillate pass over every minute. Chemists vary the method of distillation, some using 300 cc. of the oil and a larger flask of same form,

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[1] *Chem. Ztg.*, *1893*, p. 412.

[2] *Scientific American Suppl.*, 1893, p. 14733.

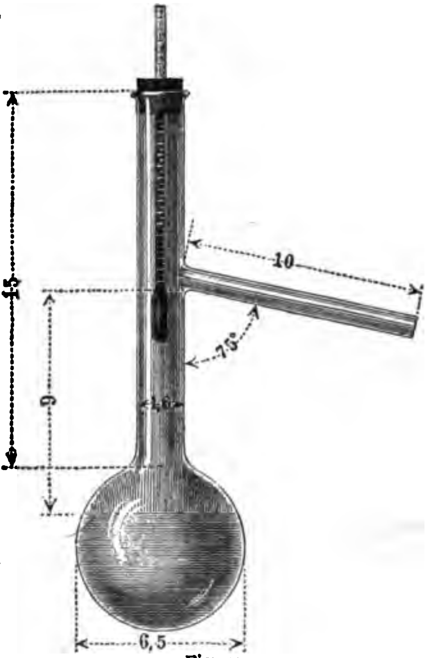

Fig. 103.

though without standard rules respecting the number of distillates to be obtained: thus A. Bourgougnon and J. Mondel[1] report the analysis of a sample of Ohio petroleum in which the distillation was in fifty parts, each part representing two per cent. by volume, the distillation commencing at 23° C. The composition of the oil being given as sixteen per cent. of naphtha, 70° B., sixty-eight per cent. of kerosene, six per cent. of paraffin oil and ten per cent. of residuum. Durand Woodman[2] gives an analysis of a crude petroleum from Ohio. 300 cc. of the oil were taken and eighteen distillates each of fifteen cc. (five per cent. of total) were obtained. The results in detail were as follows:

Number of distillate.	°F.	°B.	Per cent.
1	160	70.5	5
2	200	65.0	10
3	210	61.0	15
4	250	57.5	20
5	263	54.0	25
6	277	52.0	30
7	348	48.0	35
8	354	45.0	40
9	370	43.0	45
10	400	41.0	50
11	427	40.0	55
12	...	40.0	60
13	476	39.0	65
14	486	40.0	70
15	490	39.0	75
16	486	40.0	80
17	466	41.0	85
18	450	41.0	90
Residuum	...	...	100

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[1] *J. Am. Chem. Soc.*, 13, 168.

[2] *Ibid*, 13, 180.

The result being

Naphtha	10 per cent.
Illuminating oil	50 " "
Lubricating oil	30 " "
Residuum	10 " "
Total	100 " "

A distillation of a Mexican petroleum, by the writer, made by the Engler method, gave

Naphtha	10.0 per cent.
Illuminating oil	60.0 " "
Lubricating oil	15.5 " "
Tar and Residuum	14.5 " "
Total	100.0 " "

Another sample of the same oil, submitted to a somewhat higher temperature during the distillation, using a similar flask excepting that the delivery tube was one and one-half inches higher in the neck of the flask (requiring higher heat upon the petroleum for the same distillates as in the former case), gave a lower percentage of heavy oils, and a higher percentage in illuminating oils, the result being

Naphtha	11.0 per cent.
Illuminating oil	64.0 " "
Lubricating oil	10.3 " "
Residuum	14.7 " "
Total	100.00 " "

By a careful regulation of the heat, the amount of illuminating oil can be increased or decreased to a certain percentage as desired.

The three general divisions of the distillation of petroleum are still further technically divided as follows :

1. Naphtha group, comprises :

Cymogene, a gas, boiling point 0° C., specific gravity 110° B.
Rhigolene, liquid, boiling point 18.3° C., specific gravity 100° B.
Petroleum ether, boiling point 40° to 70° C., specific gravity 85° to 80° B.
Gasolene, boiling point 70° to 90° C., specific gravity, 80° to 75° B.
Naphtha (Danforth oil) boiling point 80° to 110° C., specific gravity 76° to 70° B.
Ligroine, boiling point 80° to 120° C., specific gravity 67° to 62° B.
Benzene, boiling point 120° to 150° C., specific gravity 62° to 57° B.

2. Illuminating oils. The various varieties of kerosene, boiling points 150° to 300° C.

3. Residuum, (tar, etc.) boiling point 300° C., and above, from which is obtained: Lubricating oils, paraffin oils, and coke remaining as a solid body in the retort.

The average percentage of the products obtained from Pennsylvania petroleum can be stated as:

First group: Naphthas, 16.5 per cent.

Second group: Illuminating oils, fifty-four per cent.

Third group: Lubricating oils, seventeen per cent., paraffin, two per cent., coke, ten per cent.[1]

The manufacture of vaseline, petrolatum, cosmoline, etc., from the tarry residuum (vacuum process,) has increased largely in the last few years.[2]

In the oil trade the principal mineral oils obtained from petroleum are as follows:

Benzenes and naphthas, 62°, 65°, 75°, 88°, 90° Baumé.

Paraffin gas oil. Paraffin oils, 22°, 24°, 25°, 28°, 30°, 32° B.

Red oil, 23° and 24° B. Neutral filtered, 32°, 34°, 37° B.

"Extra cold test" 32° B. "Wool stock" 32°. Black reduced (25° to 30° F. cold test) (15° F. cold test), 28° B. zero test. Black reduced, "Summer." Cylinder, light filtered, 600° F. fire test. Smith's ferry 32° to 34° B.

Dark steam refined. West Virginia, natural 29°; Franklin natural 29° B.

Kerosene, the different grades and colors.

The various valve oils, car oils, engine oils, spindle oils, loom oils, dynamo oils, etc., etc., are usually compounded oils, mineral oil of some variety being the principal constituent which varying amounts of lard oil, tallow oil, tallow, rape oil, etc., have been added.

The best engine oil is a mixture of lard oil and paraffin oil in equal parts. This compound has been in use by the Pennsylvania Railroad for the past ten years, and after many experiments and trials of different substitutes, still remains the standard. Passenger car oil is usually a mixture of well oil and lard

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[1] S. F. Peckham: Report on Petroleum, p. 165.

[2] Consult Brandt: Petroleum and its Products, p. 650.

oil in the proportion of two-thirds well oil and one-third lard oil. Lard oil in the proportion of one part to three of 500° well oil has been found to give the best results as a cylinder lubricator.[1]

XLIV.

The Examination of Lubricating Oils.

The generally accepted conditions of a good lubricant are as follows:

1st. Body enough to prevent the surfaces, to which it is applied, from coming in contact with each other.

2d. Freedom from corrosive acids, either of mineral, animal or vegetable origin.

3d. As fluid as possible, consistent with "body."

4th. A minimum coefficient of friction.

5th. High "flash" and "burning" points.

6th. Freedom from all materials liable to produce oxidation or "gumming."

The examinations to be made to verify the above are both chemical and mechanical, and are usually arranged in the following order:

1st. Identification of the oil, whether a simple mineral oil, animal oil, vegetable oil, or a mixture.

2d. Specific gravity.

3d. Cold test.

4th. Viscosity.

5th. Iodine absorption.

6th. Flash and fire tests.

7th. Acidity.

8th. Maumené's test.

9th. Coefficient of friction.

If the oil is a pure mineral oil, the tests numbered 1, 5 and 8 are omitted.

The first test, the nature of the oil, etc., is performed as follows:

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[1] *The Railroad and Engineering Journal*, 64, 73-126. For formulas of locomotive and car lubricants as used on the railroads in Germany consult: "Die Schmiermittel."—Von Josef Grossmann, 1894.

Ten grams of the oil are weighed out in a dry tared beaker (250 cc.), and to it is added seventy-five cc. of an alcoholic solution of potash (sixty grams of potassium hydroxide to 1,000 cc. of ninety-five per cent. alcohol), and the contents evaporated until all the alcohol is driven off. In this process, if any animal or vegetable oil is present, it is formed into a soap by the potash, while the mineral oil is unacted upon. Water (seventy-five cc.) is now added and the material well stirred to insure complete solution of the soap, and then it is transferred to a separatory funnel (Fig. 104), seventy-five cc. of sulphuric ether added, corked, the liquid violently agitated and allowed to stand for twelve hours. Two distinct liquids are now seen, the lower, the solution of the soap, the upper, the ether solution (colored, if mineral oil is present, colorless, if not). The aqueous solution is drawn off in a No. 3 beaker, the ethereal solution remaining in the separatory funnel. The former is placed on a water-bath, heated for half an hour, and until all traces of ether (which is absorbed by the water in a very small amount) is gone.

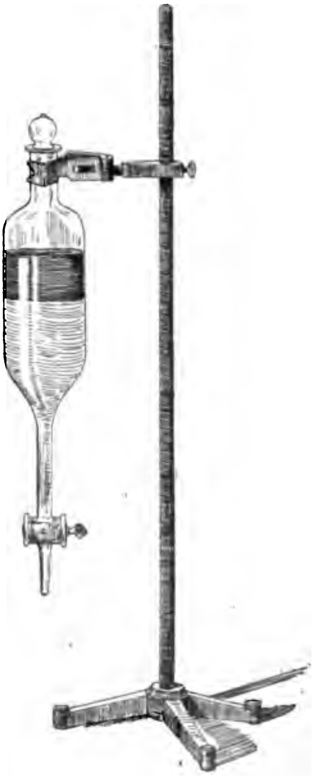


Fig. 104.

The solution is allowed to cool, diluted somewhat with water, and made acid with dilute sulphuric acid. Any animal or vegetable oil present will be indicated by a rise to the surface of the liquid of the fatty acids. (In this reaction the sulphuric acid decomposes the soap, uniting with the potash to form sulphate of potash and liberating the fatty acids of the oil.)

If it be desired to weigh the fatty acids, proceed as follows: Weigh carefully about five grams of pure white beeswax, place it in the beaker upon the surface of the oil and water, and bring the contents nearly to boiling; the melted wax and fatty acids unite; allow to cool, remove the wax, wash with water, dry between folds of filter paper and weigh. The increase in weight of the wax over its original weight gives the weight of the fatty acids of the animal or vegetable oil in the lubricating oil.

Another method of determining the weight of the fatty acids after saponification is given on page 354.

The weight obtained must be multiplied by the factor 0.97, since the fatty acids exist in the oil as anhydrides and not as hydrates, the latter being the form in which they are weighed,

Instead of weighing the animal or vegetable oil, some chemists prefer to make use of the ether solution, determining the hydrocarbon oil directly. In which case proceed as follows:

After drawing off the soap solution from the separatory funnel the ether solution is run into a weighed flask (about 250 cc.), and the ether distilled off. The residue in the flask now consists of the mineral oil and some water.

It is quite difficult to get rid of all this water. Direct heating is inadmissible, since the water spurts up through the oil out of the flask and is lost. This can be overcome by placing a glass tube through the stopper, in shape of the letter S. Any oil ejected against the tube or cork cannot escape, but returns to base of flask, while the heat is gradually increased in the flask and the water vaporized and passed out through the tube; three or four weighings are generally required before a constant weight is obtained. The former process is preferable, since it is performed much more rapidly than the latter, and also the animal or vegetable oil is positively shown, and generally can be identified; also many lubricating oils contain as high as twenty per cent. of hydrocarbon oil, volatile at or below 212° Fahrenheit. It is, of course, in the ether solution, and when the water is expelled from the oil, after the ether has been driven off, a large proportion of the volatile hydrocarbon is also vaporized. If now the animal or vegetable oil is not also determined, a serious mistake would be made; *viz.*, reporting twenty per cent. of animal oil when it was volatile mineral oil.

The fatty acids in another sample of the oil are separated and subjected to qualitative tests for identification of the oil from which they are derived. These tests comprise determination of melting point, and congealing point, page 337, color reaction with nitric and sulphuric acid, iodine absorption, and Maumené's test, rise of temperature upon addition of sulphuric acid.

There are several methods of determining the melting point ot the fatty acids. Where a considerable amount of the fatty acids is available for experiment, the apparatus in Fig. 105 can be used. The glass cylinder is filled one-half with fatty acids, the cylinder closed with a rubber stopper, through which a thermometer is inserted, the bulb of which is covered by the fatty acids.

The apparatus is supported in a beaker containing water. (Fig. 106).

If the fatty acids are liquid at ordinary temperatures, the water in the beaker must be cooled with ice until the fatty acids are congealed. The ice is removed, and the water gradually warmed until the fatty acids become melted. At this point the temperature is taken and recorded. Greater delicacy in the determination of the melting point is obtained by using a small glass tube, sealed at one end. The liquid fatty acids are placed in this tube, then congealed, the tube then tied to a thermometer(Fig. 107) and both inserted in a beaker of water, as shown in Fig. 108. Another method is to cover the thermometer bulb with a layer of the solid fatty acids, about three mm. thick and immersing it in water; gradually heat the water

Fig. 105.

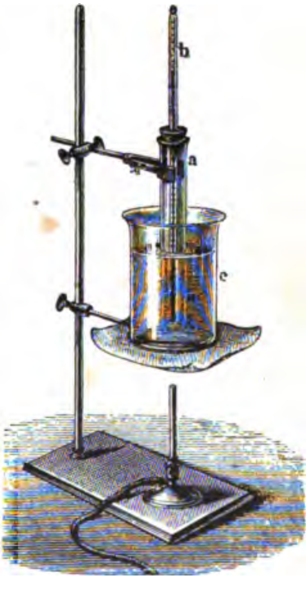


Fig. 106.

and notice the temperature at which the fatty acids leave the thermometer bulb and ascend through the water.

Fig. 107.

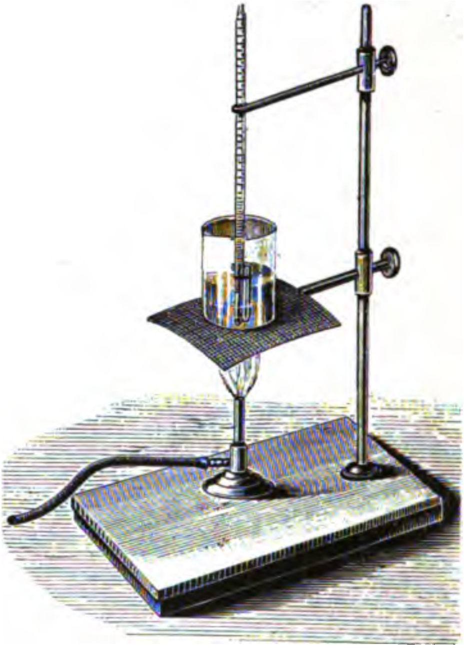


Fig. 108.

TABLE OF MELTING POINTS AND CONGEALING POINTS OF FATTY ACIDS.

Fatty acids.	Melting point.	Congealing point.
Cotton-seed oil	33.0° C.	30.5° C.
Olive "	26.0	21.0
Rape-seed "	20.0	12.0
Castor "	13.0	30.0
Sesamé "	26.0	32.0
Cocoanut "	24.5	24.0
Lard	44.0	39.0
Tallow	45.0	42.0
Wool-fat	42.0	40.0
Palm oil	48.0	43.0

Specific Gravity.

In the chemical laboratory the hydrometers used are generally marked with the specific gravity direct. In the oil trade, how-

Fig. 109.

ever, and in general commercial practice the Baumé hydrometer is used, and the following precaution is necessary.

If the oil is not liquid enough to flow easily, it must be warmed until so, and then tested with the hydrometer. The latter should move easily and freely in the liquid. As all specific gravities are comparable at 60° F., it will be necessary to make correction for temperature; if the temperature of the oil is above 60° F., the reading of the hydrometer is too large; if below 60° F., the readings are too small. Suppose an oil registers 28° Baumé at 72° F., we make use of the table, on p. 372, and find the corrected reading to be 27.2° Baumé.

To convert this into specific gravity the following table is used:

Baumé: liquids lighter than water.	Specific gravity.	Baumé: liquids lighter than water.	Specific gravity.	Baumé: liquids lighter than water.	Specific gravity.	Baumé: liquids lighter than water.	Specific gravity.
10	1.0000	23	0.9150	36	0.8433	49	0.7821
11	0.9929	24	0.9090	37	0.8383	50	0.7777
12	0.9859	25	0.9032	38	0.8333	51	0.7734
13	0.9790	26	0.8974	39	0.8284	52	0.7692
14	0.9722	27	0.8917	40	0.8235	53	0.7650
15	0.9655	28	0.8860	41	0.8187	54	0.7608
16	0.9589	29	0.8805	42	0.8139	55	0.7567
17	0.9523	30	0.8750	43	0.8092	56	0.7526
18	0.9459	31	0.8695	44	0.8045	57	0.7486
19	0.9395	32	0.8641	45	0.8000	58	0.7446
20	0.9333	33	0.8588	46	0.7954	59	0.7407
21	0.9271	34	0.8536	47	0.7909	60	0.7368
22	0.9210	35	0.8484	48	0.7865	70	0.7000

and we find that 27.2° Baumé are equal to 0.8928 specific gravity.

Figure 109 represents a Tagliabue hydrometer for oils; it contains a thermometer, also a scale to make the readings at 60° F. Subtract 1° Baumé for every 10° F. above 60° F., and add 1° Baumé for every 10° F. below 60° F.

Table of Specific Gravity with Correction for Temperature.

Indication on hydrometer.	40° F.	42° F.	44° F.	46° F.	48° F.	50° F.	52° F.	54° F.	56° F.	58° F.	60° F.	62° F.	64° F.	66° F.	68° F.	70° F.
20° B...	21.1	21.0	20.9	20.7	20.6	20.5	20.4	20.3	20.2	20.1	20.0	19.9	19.8	19.6	19.5	19.4
22° B...	23.2	23.0	22.9	22.8	22.7	22.5	22.4	22.3	22.2	22.1	22.0	21.9	21.7	21.6	21.5	21.4
24° B...	25.2	25.1	25.0	24.8	24.7	24.6	24.5	24.4	24.3	24.1	24.0	23.9	23.7	23.6	23.5	23.4
26° B...	27.3	27.2	27.0	26.9	26.7	26.6	26.5	26.4	26.3	26.1	26.0	25.9	25.7	26.6	25.5	25.3
28° B...	29.3	29.2	29.1	28.9	28.8	28.6	28.5	28.4	28.3	28.1	28.0	27.9	27.8	27.6	27.5	27.3
30° B...	31.4	31.2	31.1	31.0	30.8	30.7	30.5	30.4	30.3	30.1	30.0	29.9	29.7	29.6	29.5	29.3
32° B...	33.5	33.3	33.1	33.0	32.9	32.7	32.6	32.4	32.3	32.1	32.0	31.9	31.7	31.6	31.4	31.3
34° B...	35.5	35.4	35.2	35.1	34.9	34.8	34.6	34.5	34.3	34.2	34.0	33.8	33.7	33.5	33.4	33.3
36° B...	37.6	37.4	37.2	37.1	36.9	36.8	36.6	36.5	36.3	36.2	36.0	35.8	35.7	35.5	35.4	35.2
38° B...	39.6	39.4	39.3	39.1	39.0	38.8	38.6	38.5	38.3	38.2	38.0	37.8	37.7	37.5	37.4	37.2
40° B...	41.6	41.5	41.3	41.2	41.0	40.8	40.7	40.5	40.3	40.2	40.0	39.8	39.7	39.5	39.4	39.2
42° B...	43.7	43.5	43.4	43.2	43.0	42.9	42.7	42.5	42.3	42.2	42.0	41.8	41.7	41.5	41.3	41.2
44° B...	45.7	45.6	45.4	45.2	45.1	44.9	44.7	44.5	44.4	44.2	44.0	43.8	43.7	43.5	43.3	43.1
46° B...	47.8	47.6	47.4	47.3	47.1	46.9	46.7	46.6	46.4	46.2	46.0	45.8	45.7	45.5	45.3	45.1
48° B...	49.9	49.7	49.5	49.3	49.1	48.9	48.8	48.6	48.4	48.2	48.0	47.8	47.6	47.4	47.3	47.1
50° B...	52.0	51.8	51.6	51.4	51.2	51.0	50.8	50.6	50.4	50.2	50.0	49.8	49.6	49.4	49.2	49.1
52° B...	54.0	53.8	53.6	53.4	53.2	53.0	52.8	52.6	52.4	52.2	52.0	51.8	51.6	51.4	51.1	51.0
56° B...	58.2	58.0	57.8	57.5	57.3	57.1	56.9	56.7	56.4	56.2	56.0	55.8	55.6	55.4	55.2	55.0
58° B...	60.3	60.0	59.8	59.6	59.3	59.1	58.9	58.7	58.4	58.2	58.0	57.8	57.6	57.3	57.1	56.9
60° B...	62.3	62.1	61.9	61.6	61.4	61.1	60.9	60.7	60.5	60.2	60.0	59.8	59.6	59.3	59.1	58.9
64° B...	66.5	66.2	66.0	65.7	65.5	65.2	65.0	64.7	64.5	64.2	64.0	63.8	63.5	63.3	63.1	62.8
66° B...	68.6	68.3	68.0	67.8	67.5	67.2	67.0	66.8	66.5	66.3	66.0	65.7	65.5	65.3	65.0	64.8
70° B...	72.7	72.5	72.2	71.9	71.6	71.3	71.1	70.8	70.5	70.3	70.0	69.7	69.5	69.2	69.0	68.7
72° B...	74.8	74.5	74.2	73.9	73.6	73.3	73.1	72.8	72.5	72.3	72.0	71.7	71.5	71.2	70.9	70.7
76° B...	78.9	78.6	78.3	78.0	77.7	77.4	77.1	76.9	76.6	76.3	76.0	75.7	75.4	75.2	74.9	74.6
80° B...	83.0	82.7	82.4	82.1	81.8	81.5	81.2	80.9	80.6	80.3	80.0	79.7	79.4	79.1	78.8	78.5

Table of Specific Gravity with Correction for Temperature (*Continued*).

Indication on hydrometer.	72° F.	74° F.	76° F.	78° F.	80° F.	82° F.	84° F.	86° F.	88° F.	90° F.	92° F.	94° F.	96° F.	98° F.	100° F.
20° B..........	19.3	19.2	19.1	19.0	18.9	18.7	18.7	18.6	18.5	18.4	18.2	18.1	18.0	17.8	17.9
22° B..........	21.3	21.1	21.0	20.9	20.8	20.7	20.6	20.5	20.4	20.3	20.2	20.1	19.9	19.8	19.7
24° B..........	23.2	23.1	23.0	22.9	22.8	22.6	22.5	22.4	22.3	22.2	22.1	21.9	21.8	21.7	21.6
26° B..........	25.2	25.1	25.0	24.8	24.7	24.6	24.5	24.4	24.2	24.1	24.0	23.9	23.7	23.6	23.5
28° B..........	27.2	27.1	26.9	26.8	26.7	26.5	26.4	26.3	26.2	26.0	25.9	25.8	25.6	25.5	25.4
30° B..........	29.2	29.0	28.9	28.8	28.6	28.5	28.4	28.2	28.1	28.0	27.8	27.7	27.6	27.5	27.3
32° B..........	31.2	31.0	30.9	30.7	30.6	30.4	30.3	30.2	30.0	29.9	29.7	29.6	29.5	29.3	29.2
34° B..........	33.1	36.0	32.8	32.7	32.5	32.4	32.2	32.1	32.0	31.8	31.6	31.5	31.4	31.2	31.1
36° B..........	35.1	34.9	34.8	34.6	34.5	34.3	34.2	34.0	33.9	33.7	33.5	33.4	33.3	33.1	33.0
38° B..........	37.1	36.9	36.8	36.6	36.4	36.3	36.1	36.0	35.8	35.6	35.5	35.3	35.2	35.0	34.9
40° B..........	39.0	38.9	38.7	38.5	38.4	38.2	38.1	37.9	37.7	37.6	37.4	37.2	37.1	36.9	36.8
42° B..........	41.0	40.8	40.7	40.5	40.3	40.2	40.0	39.8	39.7	39.5	39.3	39.2	39.0	38.9	38.7
44° B..........	43.0	42.8	42.6	42.5	42.3	42.1	42.0	41.8	41.6	41.4	41.3	41.1	40.9	40.8	40.6
46° B..........	44.9	44.8	44.6	44.4	44.2	44.1	43.9	43.7	43.6	43.4	43.1	43.0	42.9	42.7	42.6
48° B..........	46.9	46.7	46.5	46.4	46.2	46.0	45.8	45.7	45.5	45.3	45.2	45.0	44.8	44.6	44.5
50° B..........	48.9	48.7	48.5	48.3	48.1	48.0	47.8	47.6	47.4	47.3	47.1	46.9	46.7	46.5	46.4
52° B..........	50.8	50.6	50.4	50.3	50.1	49.9	49.7	49.5	49.4	49.2	49.0	48.8	48.6	48.4	48.3
56° B..........	54.8	54.5	54.3	54.1	53.9	53.7	53.5	53.4	53.2	53.0	52.8	52.6	52.4	52.2	52.0
58° B..........	56.7	56.5	56.3	56.1	55.9	55.7	55.5	55.3	55.1	54.9	54.7	54.5	54.3	54.1	53.9
60° B..........	58.7	58.5	58.2	58.0	57.8	57.6	57.4	57.2	57.0	56.8	56.6	56.4	56.2	55.9	55.7
64° B..........	62.6	62.4	62.1	61.9	61.7	61.4	61.2	61.0	60.8	60.6	60.4	60.2	59.9	59.7	59.5
66° B..........	64.5	64.3	64.1	63.8	63.6	63.4	63.2	62.9	62.7	62.5	62.3	62.1	61.8	61.6	61.4
70° B..........	68.4	68.2	68.0	67.7	67.5	67.2	67.0	66.7	66.5	66.3	66.1	65.8	65.6	65.3	65.1
72° B..........	70.4	70.2	69.9	69.6	69.4	69.2	68.9	68.7	68.4	68.2	68.0	67.7	67.4	67.2	67.0
76° B..........	74.3	74.0	73.8	73.5	73.2	73.0	72.7	72.5	72.2	71.9	71.7	71.4	71.2	70.9	70.7
80° B..........	78.2	77.9	77.6	77.3	77.1	76.8	76.5	76.3	76.0	75.7	75.5	75.2	74.9	74.6	74.4

Thus, if the hydrometer, when placed in the oil, reads 26° Baumé and the temperature of the oil 80° F., the correct reading will be 24° Baumé at 60° F. The specific gravity test is an important one; by it an admixture of certain oils with mineral oil is indicated. For instance, a lubricating oil of specific gravity 0.915 was found by qualitative analysis to be composed of mineral oil and menhaden oil. Knowing the kinds of oil composing the mixture, an approximation of the per cents. would be obtained as follows:

Mineral oil........	Specific gravity	= 0.890 (B)
Menhaden oil.....	" "	= 0.927 (A)
Specific gravity of mixture		= 0.915 (M)

Let $A - M = C$. $(0.927 - 0.915 = 0.012)$
$M - B = D$. $(0.915 - 0.890 = 0.025)$

$$\text{Then } \frac{D}{C+D} = \text{per cent. of } A\left(\frac{0.025}{0.037}\right),$$

and

$$\frac{C}{C+D} = \text{per cent. of } B\left(\frac{0.012}{0.037}\right).$$

The result being

Menhaden oil	67.5 per cent.
Mineral "	32.5 " "

A more rapid method is graphically thus: in Fig. 110 let the abscissas represent per cents. and the ordinates the specific gravities. From the point indicated (on the line *A*—*B*) 0.915 the specific gravity of the mixture the per cents. are read on abscissa line 67.5 for *A* and 32.5 per cent for *B*.

Another instrument used for the determination of the specific gravity of oils is the Westphal balance.

This apparatus (Fig. 111) is very accurate and should be used as a check determination of the gravity made by the hydrometer.

If the oil is too thick, at ordinary temperatures, for the determination of the gravity, it should be heated sufficiently and the modified Westphal balance (Fig. 112) used.

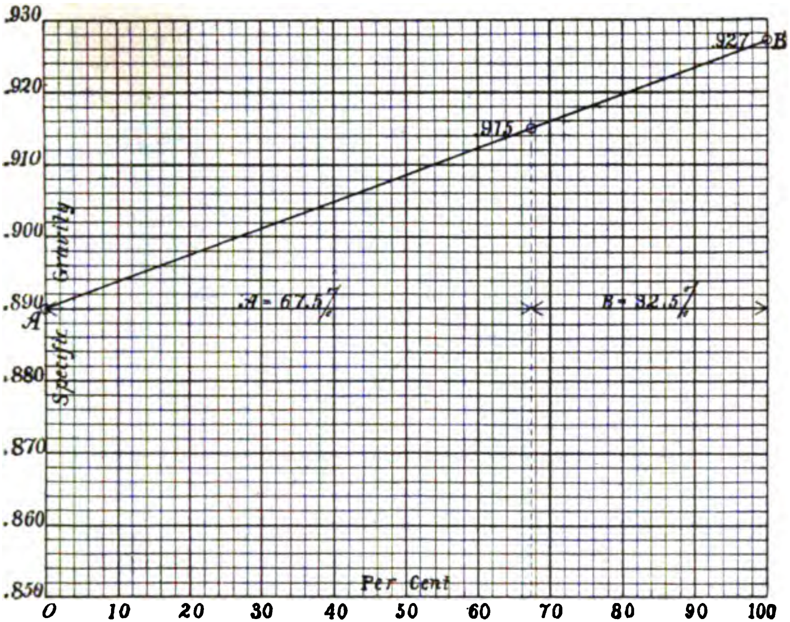


Fig. 110.

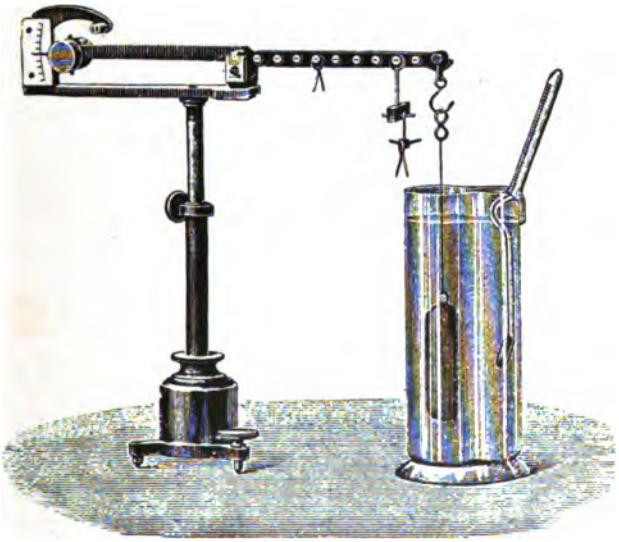


Fig. 111.

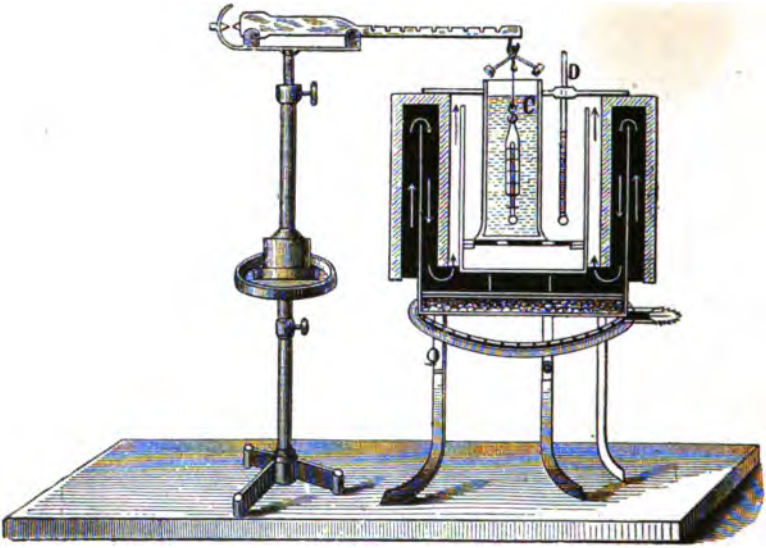


Fig. 112.

If only small amounts of the oil are obtainable a small picnometer, or an Araeo-picnometer of Eichhorn can be used. This invention is described by Dr. H. Hensoldt, of the Petrographical Laboratory of Columbia College, New York, in the "*Scientific American Supplement*" of March 21, 1891, with a drawing. The important feature of this instrument consists in a small glass bulb (attached to the spindle), which is filled with the liquid whose gravity is to be taken. Thus instead of floating the entire apparatus in the test fluid, only a very small quantity of the latter is required. (Fig. 113.)

The glass bulb, when filled with the test fluid, is closed by means of an accurately filling glass stopper, and the instrument is then placed in a glass cylinder filled with distilled water at 17.5° C.

The gravity is then at once shown on the divided scale in upper portions of the spindle.

The following table converts degrees of the various hydrometers into specific gravity. (Liquids lighter than water.)

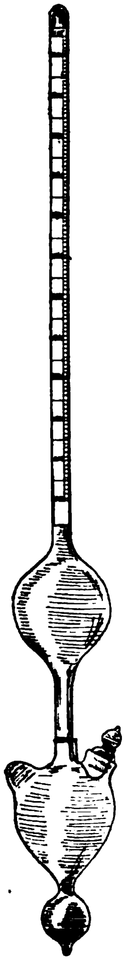


Fig. 113.

$$\text{Gay-Lussac, } 4^\circ \text{ C} = \frac{100}{100 + n} = \text{specific gravity.}$$

$$\text{Beck, } 12.5^\circ \text{ C.} = \frac{170}{170 + n} = \text{specific gravity.}$$

$$\text{Cartier, } 12.5^\circ \text{ C.} = \frac{136.8}{126.1 + n} = \text{specific gravity.}$$

$$\text{Baumé hydrometer, at } 15^\circ \text{ C.} = \frac{144.78}{134.78 + n} = \text{sp. gr.}$$

$$\begin{array}{l}\text{Brix hydrometer, Fischer} \\ \text{hydrometer at } 15.6^\circ \text{ C.}\end{array} = \frac{400}{400 + n} = \text{sp. gr.}$$

n = degrees indicated upon the spindle.

TABLE OF SPECIFIC GRAVITY OF OILS USED WITH MINERAL OILS FOR LUBRICATING PURPOSES.

Sperm oil	0.883	specific	gravity.
Olive oil	0.916	"	"
Cotton-seed oil (white)	0.925	"	"
Cotton-seed oil (brown)	0.930	"	"
Castor oil	0.960	"	"
Dolphin oil	0.922	"	"
Neat's foot oil	0.915	"	"
Lard oil	0.915	"	"
Tallow oil	0.903	"	"
Menhaden oil	0.928	"	"
Rape-seed oil	0.916	"	"
Rosin oil	0.980 to 1.05	"	"
Blown oils, made by oxidation of rape-seed oil, cotton-seed oil, etc., (consult Chapter 46)	0.930 to 0.970	"	".

References on the specific gravity of oils:

"On Fluid Specific Gravity Determinations for Practical Purposes." By C. R. Alder Wright, F.R.S., *J. Soc. Chem. Ind.*, 11, 297.

"On the Chemistry and Analytical Examination of Fixed Oils." By Alfred H. Allen, F.C.S., *J. Soc. Chem. Ind.*, 2, 49–59; 6, 65–73.

The Cold Test.

The degree at which an oil becomes semi-solid and refuses to flow freely is considered the cold test, and is performed as follows:

Fifty cc. of the oil are transferred to a narrow bottle (capacity

100 cc.), stoppered with a rubber stopper, through which is inserted a thermometer, the bulb of which reaches an inch or more into the oil.

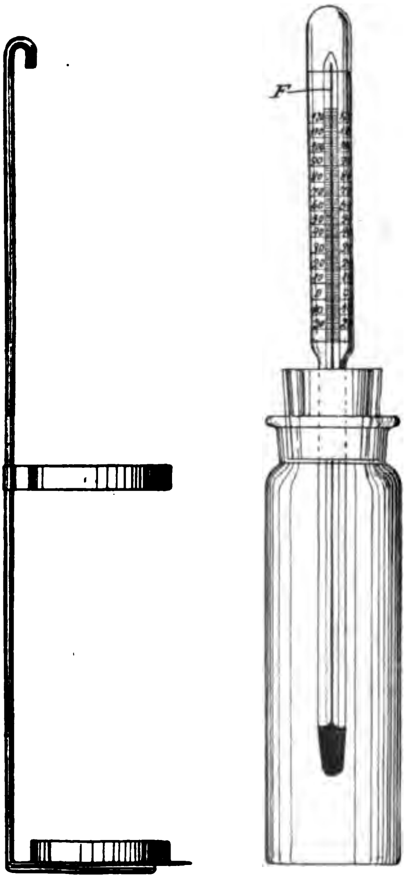


Fig. 114.

The bottle is placed in a mixture of ice and salt, or other freezing compound, and retained there until the oil becomes solid. It is then removed and allowed to warm until the contents become somewhat thinner in consistence. The bottle is inclined from side to side until the oil begins to flow, when the temperature is taken.

At this particular temperature the oil is neither at its normal fluidity, nor is it solid, and while this method does not correctly indicate the exact temperature of the solidifying point, it does show the point at which the oil ceases to flow readily, the important one to the oil inspector.

In lubricating oils, to be used in railroad practice, this cold test is a vital one, and receives in the laboratories of the different railroads of the United States considerable attention.

A mineral lubricating oil, non-paraffin, of good quality, does not show any material difference in its consistency at 25° C. or 10° C., but a radical change would be indicated at 10° C. if some of the animal or vegetable oils were a component.

While it is true that no proportion of one or the other can be indicated by the cold test, and that this test might not properly be classed as a chemical, but rather as a physical one, yet so important is this property of congealing in lubrication, and as

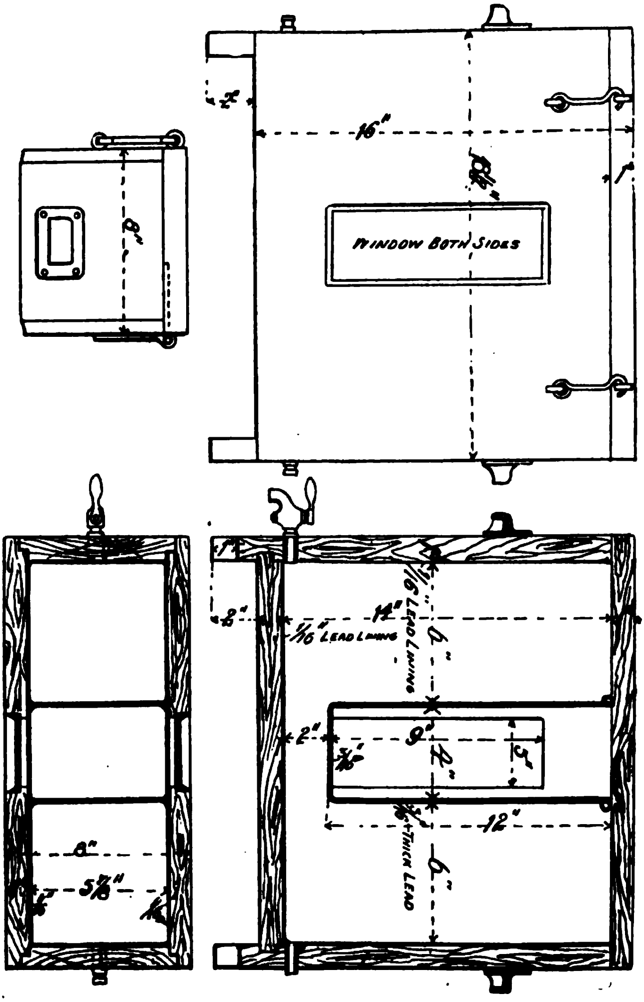


Fig. 115.

all laboratories connected with railroad work rely strongly upon it, it is included as one of the principal ones.

In connection therewith is here included the drawings of the apparatus used for this purpose in the chemical laboratory of the

Chicago, Burlington and Quincy Railroad Co., Aurora, Ill.

Fig. 114 represents the glass apparatus with the thermometer arranged for the cold test.

Fig. 115 represents the cold box to contain the freezing mixture and in which the oil is tested.

The following determinations of the cold test, made in my laboratory, will show the wide range in this regard between many of the oils, used in lubrication :

Elain oil	6° C.
Saponified red oil	5
Prime neat's foot oil	4
White neat's foot oil	4
Pure hoof oil	6
Prime lard oil	7
No. 1 lard oil	7
XXX lard oil	3
American sod oil	1
English sod oil	24
Tallow oil	26
Dog fish oil	7
Right whale oil (Pacific)	0
Unbleached bowhead whale oil (Pacific)	7
Bleached whale oil (Pacific)	13
Natural sperm oil (Pacific)	0
Bleached sperm oil "	4
Herring oil "	0
Natural winter sperm oil (Atlantic)	1
Bleached winter sperm oil "	4
Natural spring sperm oil "	10
Bleached spring sperm oil "	8
Natural winter whale oil "	2
Bleached winter whale oil "	5
Natural spring whale oil "	5
Bleached spring whale oil "	2
Prime crude menhaden oil	4
Brown strained menhaden oil	7
Light strained menhaden oil	7
Natural winter menhaden oil	9
Bleached winter menhaden oil	12
Extra bleached winter white menhaden oil	11
Bank oil	4
Straits oil	7
Sea elephant oil	5

Black fish oil	8° C.
Rosin oil, 1st run	3
" " 2d run	19
" " 3d run	20
Castor oil	18
Crude cotton-seed oil	7
Prime summer yellow cotton-seed oil	5
Off quality summer yellow cotton-seed oil	6
Prime quality winter cotton-seed oil	10
Off quality winter cotton-seed oil	8
Prime quality summer white cotton-seed oil	3
Off quality summer white cotton-seed oil	8
Prime quality winter white cotton-seed oil	9
Off quality winter white cotton-seed oil	5
No. 1 French Degras oil	25
No. 2 " " "	25
English Degras oil	18
Olive oil	3
Oleo oil	24

In the specifications, for the supply of oils to the various railroads, it is generally stated what degree is required for the cold test. Thus the Pennsylvania Railroad Co. requires as follows:

Lard oil 8° C. November 1 to April 1.
Tallow oil, 8° C. " " " "
Neat's foot oil, 8° C. " " " "

Baltimore & Ohio Railroad Co:

Engine oil from October 1 to May 1, below 9° C.
Passenger car oil " " " " " " " "
Freight car oil " " " " " " " "

Chicago, Burlington & Quincy Railroad Co. Black Engine oils:

Summer oil must flow at 15° C. and above.
25° oil " " " 1° C. " "
15° oil " " " 9° C. " "
Zero oil " " " 15° C. " "

Tagliabue's standard lubricating oil freezer is also largely used in this connection, and is thus described. It consists of a semicylindric metallic stand, neatly japanned, divided into three compartments. (Apparatus is shown in Figs. 91 and 116).

The first, *f*, is the oil cooling chamber, in which is the glass receiver, adjusted to a rocking shaft, *g*, to facilitate the introduction of the regulation oil cup therein, and to show by its motion whether the oil is congealing or not.

The second, *c*, is the ice chamber which is filled with ice and rock salt for the cooling process; a faucet, *h*, is connected with it, to allow the melted ice to flow out. The third, *a*, is a non-conductor jacket, lined with mineral wool filling, to maintain an even temperature in the cooling chamber, and to prevent a too rapid melting of the ice.

Three thermometers, *d*, are inserted in the freezer, one on

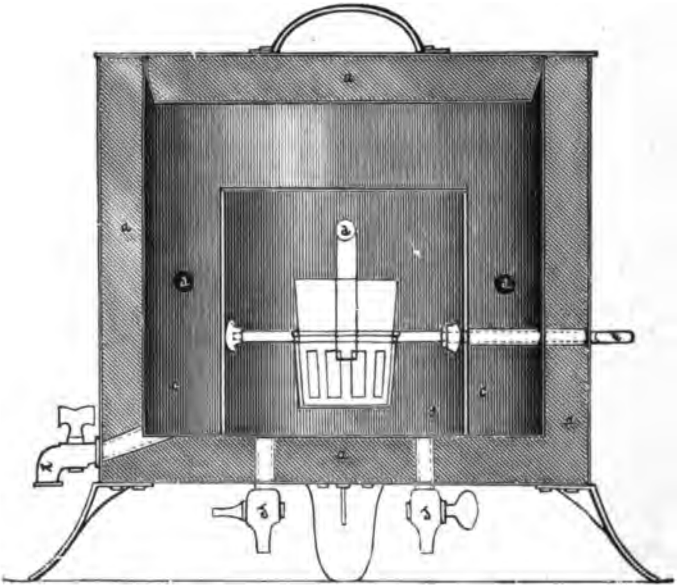


Fig. 116.

each side of the cooling chamber, to denote its temperature and a third one in the center so adjusted that its bulb, penetrating the middle of the oil, enables one to see through the glass door, *k*, (without opening the same,) at what temperature the oil congeals.

Two stop-cocks, *j*, are attached to the bottom, with the cooling chamber, to force therein (by either opening or blowing through them with a rubber tube) atmospheric or warm air, whenever it is desired to raise its temperature.

Viscosity.

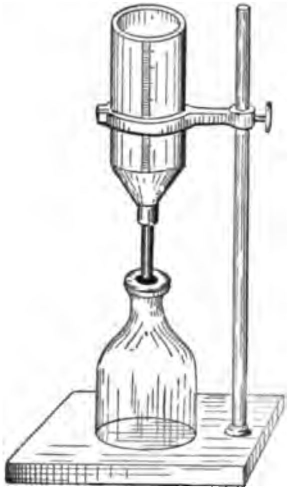


Fig. 117.

The first instrument for the determination of the viscosity of oils was probably Schubler's. (Fig. 117). It consisted of a glass cylinder, open at the top and drawn to a one thirty-second inch tube at the bottom. Having filled the cylinder with the oil to be tested, the time required for 100 cc. of the oil to flow out through the aperture was noted, and this figure compared with that obtained from water under similar conditions.

Thus, Schubler records, among many determinations, the following:

	Seconds at 15° C.	Seconds at 7.5° C.	Comparative thickness with water at 15° C	Comparative thickness with water at 7.5° C.
Colza oil	162.0	222.0	18.0	22.4
Olive oil	195.0	284.0	21.6	31.5
Hemp-seed oil.........	87.0	107.0	9.6	11.9
Castor oil	1830.0	3390.0	203.0	377.0
Distilled water	9.0	9.0	0.0	0.0

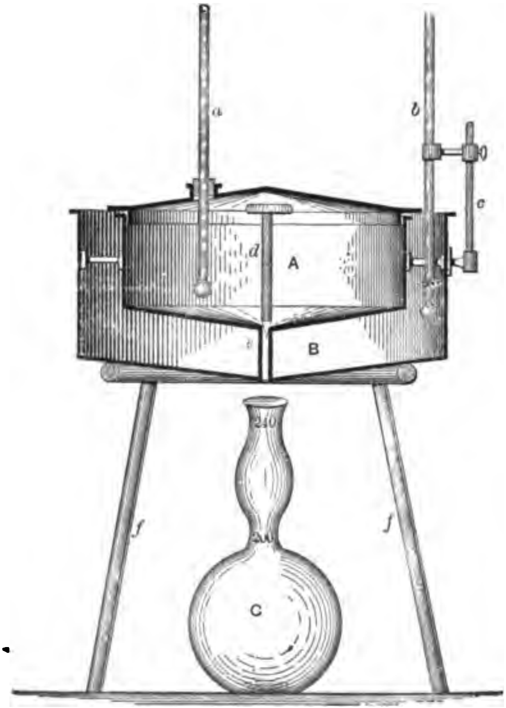
The Pennsylvania Railroad Co. viscosity tests are made as follows:

A 100 cc. pipette of the long bulb form is regraduated to hold just 100 cc. to the bottom of the bulb. The size of the aperture at the bottom is then made such that 100 cc. of water at 100° F. will run out of the pipette down to the bottom of the bulb in thirty-four seconds.

Pipettes with bulbs varying from one and three-fourths inches to one and one-half inches in diameter outside, and about four and one-half inches long, give almost exactly the same results, provided the aperture at the bottom is the proper size. The pipette being obtained, the oil sample is heated to the required temperature, care being taken to have it uniformly heated, and then is drawn up into the pipette to the proper mark. The time occupied by the oil in running out, down to the bottom of the bulb gives the test figures. A stop watch is convenient, but not essential, in making the test. The temperature of the room affects the test a little. The limiting figures were obtained in a room at from 70° to 80° F. It will not usually be possible to make duplicate tests without readjustment of the temperature of the oil.

These pipettes are in use in many railroad laboratories in the United States, but are difficult to clean, and are not as convenient as the Engler or Redwood viscosimeters.

Engler's viscosimeter (original form, Fig. 118) is constructed of copper, and consists of *A*, a chamber holding the oil to be tested; *B*, the water bath, *C*, a flask graduated so as to receive 200 cc. of the oil; *a*, *b*, thermometers; *e* the opening through which the heated oil flows out upon the withdrawal of the plug *d*.



Am. Bank Note Co. N.Y.

Fig. 118.

In using this instrument the viscosity of an oil is stated in seconds required for 200 cc. of the oil to run into the flask *C*. Heat can be applied to the water-bath, the viscosity being determined at any temperature required up to 100° C. Any temperature up to 360° C. can be secured by filling *B* with paraffin instead of water.

Engler recommends that all viscosity be compared with water thus:

If water requires 52 seconds for delivery of 200 cc. into the receiving flask, and an oil under examination requires 130 seconds,

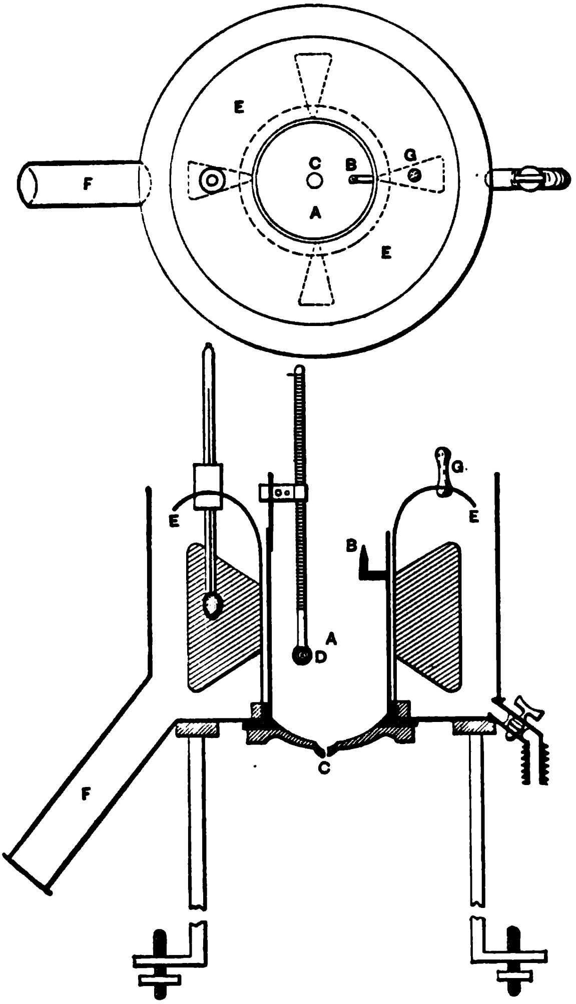


Fig. 119.

the ratio is determined by $\frac{130}{52} = 2.50$, the oil thus having a viscosity of 2.5 times that of water.

This instrument has been for many years the standard in Germany.

Boverton Redwood[1] describes a viscosimeter (Fig. 119), the general principle of which is the same as Engler's. This is the standard viscosimeter for the English oil trade.

The septometer (Figs. 120, 121), originated with Dr. Lepenau, is used for the direct comparison of the viscosity of two oils under similar conditions at the same moment. It consists of two cylindrical vessels, *B*, *B*, which hold the oils to be compared, and which stand in the same water bath, *A*, and have the same temperature. To use the apparatus the holder, *A*, is filled with water, which can be heated at any temperature desired below 100° C.; if higher temperatures are desired, *A* must be filled with oil. The vessel, *B*, is filled with the oil which is taken for the standard, such as rape oil or lard oil, and the second one is filled with the oil to be tested. Since the heated or cooled water is stirred regularly the oils have the same temperatures which are read from the thermometers, *t*, *t*. For comparison the oils are allowed to flow out, at the same time, for the same length of time. The relative value sought is found then by measuring or weighing the amounts which have flowed out.

Fig. 120.

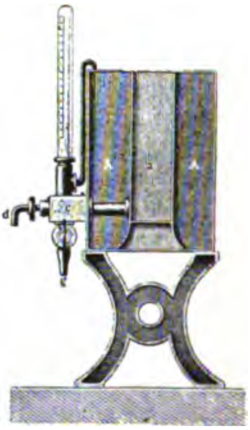


Fig. 121.

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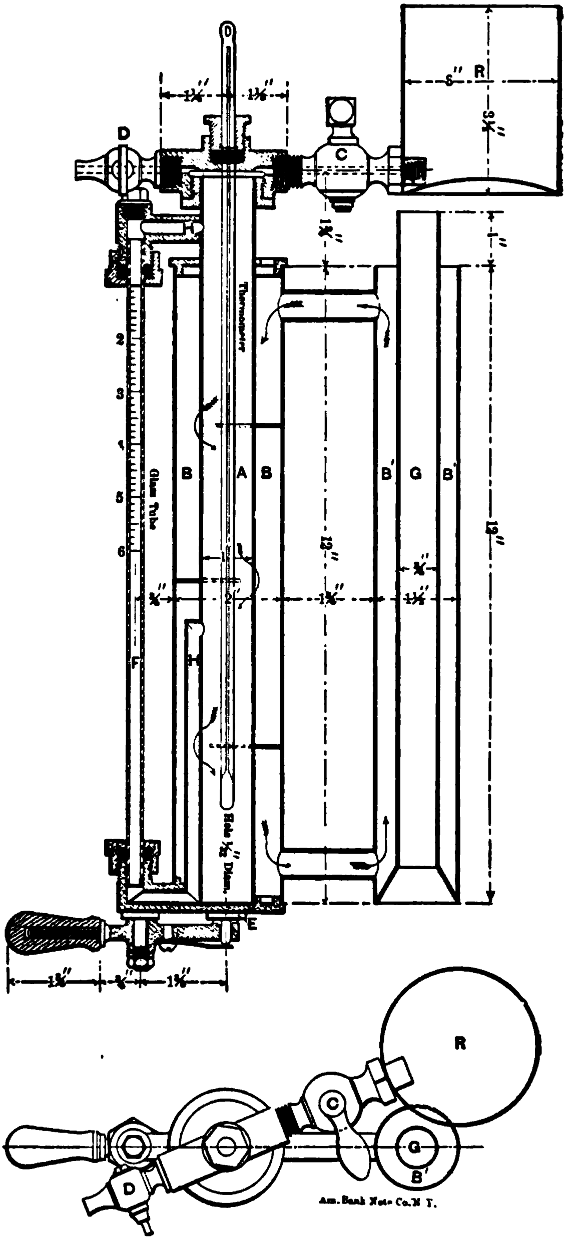
[1] *J. Soc. Chem. Ind.*, 5, 158.

Davidson's viscosimeter (Fig. 122) is designed especially for determining the relative viscosity of oils and greases when heated to the temperature of locomotive cylinders (250° to 375° F.). The entire apparatus, except the glass portion, is made of copper and the joints brazed.

The oil to be tested is put into the cylinder, *A*, and the cup, R, which are connected through the stop-cock *C*. The cylinder, *A*, is also connected with the glass gauge through the tubes, *H*, and *H*, so that the height of the oil in the cylinder can be seen. The bottom of cylinder *A* is covered by a brass plate, through which is bored a hole three and one-half inches in diameter, which can be closed by the slide valve, *E*, against the plate by a spring. The outside of the plate is beveled from the hole, so that the hole is in a very thin plate, and thus lateral friction is reduced to a minimum. A long thermometer is used, so that the bulb will be near the bottom of cylinder *A*.

The cylinders, *B* and *B'*, contain the lard oil bath that is used for conveying heat to the oil in cylinder *A*. Heat is applied by lamp or gas burner at the base of cylinder *B'*, and the hot products of combustion allowed to pass through the cylinder *G*. As the lard oil in *B'* becomes heated, it rises to the top of this cylinder, and passes over to cylinder *B*, down *B*, passing around the cylinder *A*, and back to *B'*, where it is reheated and recirculated, as shown by the arrows. The oil in cup *R* is heated by the products of combustion escaping from the top of cylinder *G*, and in case of a high temperature by an additional lamp placed under the cup *R*.

When the oil under test in *A* and *R* has reached the desired temperature, the valve, *E*, is opened and the stop-cock *C* is adjusted to keep the height of oil in *A* the height desired, as shown by the glass gauge. A 100 cc. flask, which is immersed in hot oil, is then placed under the stream of oil flowing from the hole, and a stop-watch is started the instant the oil commences to run into the flask. When 100 cc. have been delivered into the flask, the watch is stopped. The number of seconds required for this is the viscosity of the oil under examination.



Am. Bank Note Co. N. Y.

Fig. 122.

Tagliabue's vicosimeter (Fig. 123), consists of a copper basin, *C*, extending by means of the coiled tube to the outlet at the stopcock on the outside of the vessel.

This is surrounded by the water bath, *B*, which has an outer chamber *a* connected by two tubes, and in which the water is poured into the bath. *D* is a thermometer, and records the temperature of the water-bath.

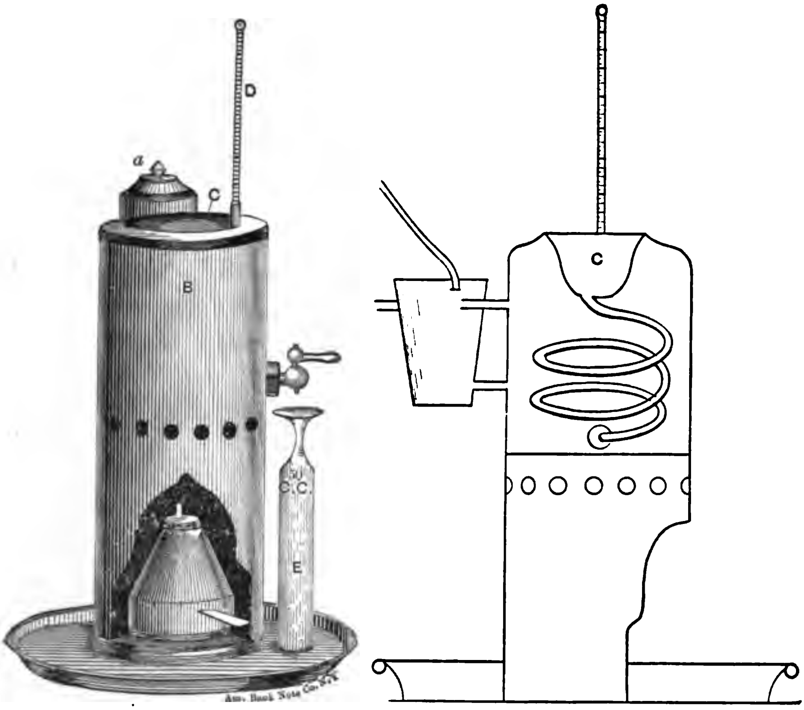


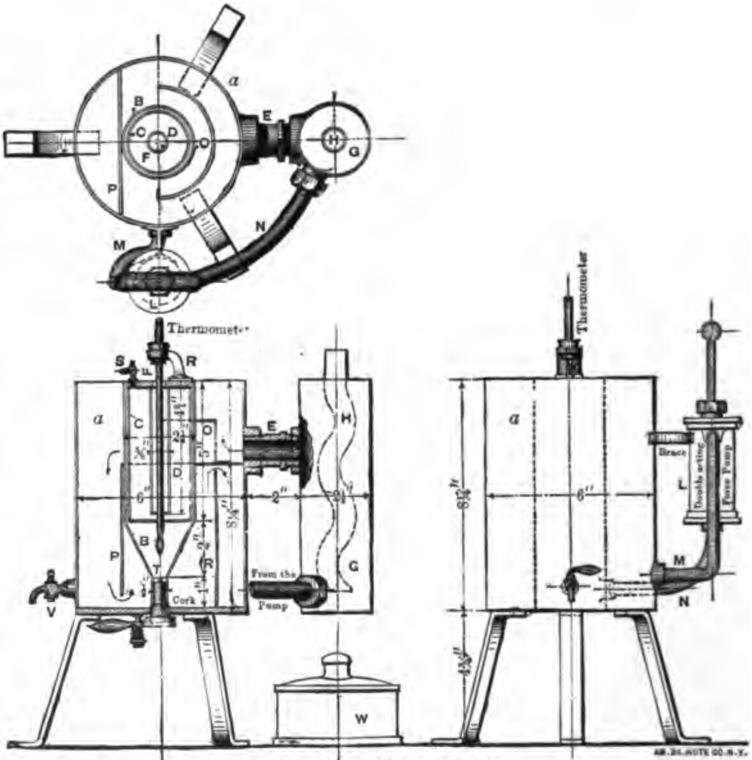
Fig. 123.

To test an oil, the water-bath is filled two-thirds full and heated by means of a small Bunsen burner or alcohol lamp. The top basin, *C*, lined with wire gauze is filled with the oil to be tested, and when the thermometer, *D*, indicates 100° C., the glass measuring flask, *E*, is placed under the faucet, which is opened with the starting of the watch.

When fifty cc. of the oil have run out and reached the mark upon the neck of the receiving flask, *E*, the watch is stopped, and the number of seconds required noted.

The viscosity of the oil is stated in seconds.

This viscosimeter has a very extended use in the oil trade but it is a difficult piece of apparatus to clean when any particles of dirt have become lodged in the coil. This materially interferes with the flow of oil through the tube and gives false results. The basin, *C*, as well as the coil, cannot be removed, as they are brazed to the water-bath.



C. M. & ST. PAUL RY. CO.
(Motive Power Dept.)
GIBBS' VISCOSIMETER.
Fig. 124.

For this reason, and also when used at higher temperatures, the faucet being metallic and not heated to the temperature of the oil, the oil leaves the apparatus much cooler than the temperature recorded by the thermometer of the water-bath.

Gibb's viscosimeter, Fig. 124 (George Gibbs, M. E., Chicago, Milwaukee and St. Paul Railroad), was designed to overcome some objectionable points in existing forms of viscosimeters.

The idea being: *First.*—To have a large body of hot oil as a bath surrounding the oil to be tested in order to keep the latter at a perfectly uniform temperature.

Second.—To apply a forced circulation to the bath by means of a double action pump, to insure equality of heat in all parts.

Third.—To deliver the oil to be tested at the orifice under a constant head, which is accomplished by means of a pneumatic trough.

Fourth.—To supply convenient means for accurately measuring the temperature of the oil near its delivery point.

The large reservoir *a* is of copper, with heavy brazed bottom. This contains the cylindrical inside chamber with conical bottom, *B*. At the lower end of this is the gauged aperture, *T*. Inside of this chamber fits the inverted reservoir, *C*, holding the oil to be tested. In the interior of this chamber is a tube, *D*, extending nearly to the bottom of the same. This tube admits air to determine the head of the oil, and also to admit the thermometer, *F*. The outside bath, *a*, contains the deflector plates, *O*, *P* and *R* to obtain proper mixing of the bath. The heating of the bath is done by a lamp, *W*, set underneath the separate heating chamber, *G*. The size of the orifice at *T* is one-sixteenth inch.

The following table shows the result of viscosity tests upon various oils made with this instrument.

VISCOSITIES OF VALVE OILS AND STOCKS.

	Gravity.	Flash. F.	Per cent. mineral oil.	Viscosities			
				250° F.	300° F.	350° F.	400° F.
Nat. Ref'g Co., Loco. Cyl..................	26.8	525°	75.7	38 sec.	32	26	......
Nat. Ref'g Co., German	25.8	550	7.00	43	33	28	......
Perfection valve oil...	26.0	510	54.7	35	29	25	......
" "(another)	25.7	undet	65.0	34	28	24	......
" " "	25.9	510	undet.	......	......	23	21
Vacuum valve oil.....	25.2	535	95.0	......	.....	27	23
C., M. & St. P. valve oil	26.4	485	66.7	......	......	23	21
Extra lard oil (average of 3 samples).......	......	......	......	25	23	21	20
St'd Oil Co., No. 1 stock	27.0	520	100	46	32	26	22
" " 2 "	27.3	510	100	47	32	26	22
" " 3 "	27.8	490	100	39	30	25	21
" " 4 "	26.2	525	100	46	33	27	23

Viscosities expressed in seconds for 50 cc.

VISCOSITIES OF CAR AND ENGINE OIL.

	Gravity.	Flash. F.	Per cent. mineral oil.	Viscosities.		
				75° F.	110° F.	150° F.
National Ref'g Co., car oil...	30.8	200°	100	223	68	41
Relief Oil Works, " ...	30.4	200	100	163	61	38
Galena car oil...............	28.5	160	90	102	54	36
" " "...............	28.2	165	90	83	50	33
" " "...............	28.7	155	90	102	54	36
" " ".	27.8	170	90	88	52	34
" " "...............	26.3	285	91.9	234	99	49
" " "...............	26.5	260	91.0	257	98	48
Relief Oil Works, engine oil.	30.1	210	100	130	64	37
National Ref'g Co., " ".	26.5	385	100	740	113	54

Viscosities expressed in seconds for 50 cc.

The viscosities of a number of other oils, at the temperature of locomotive cylinders, as made by this instrument, are shown in the chart of curves. (Fig. 125.)

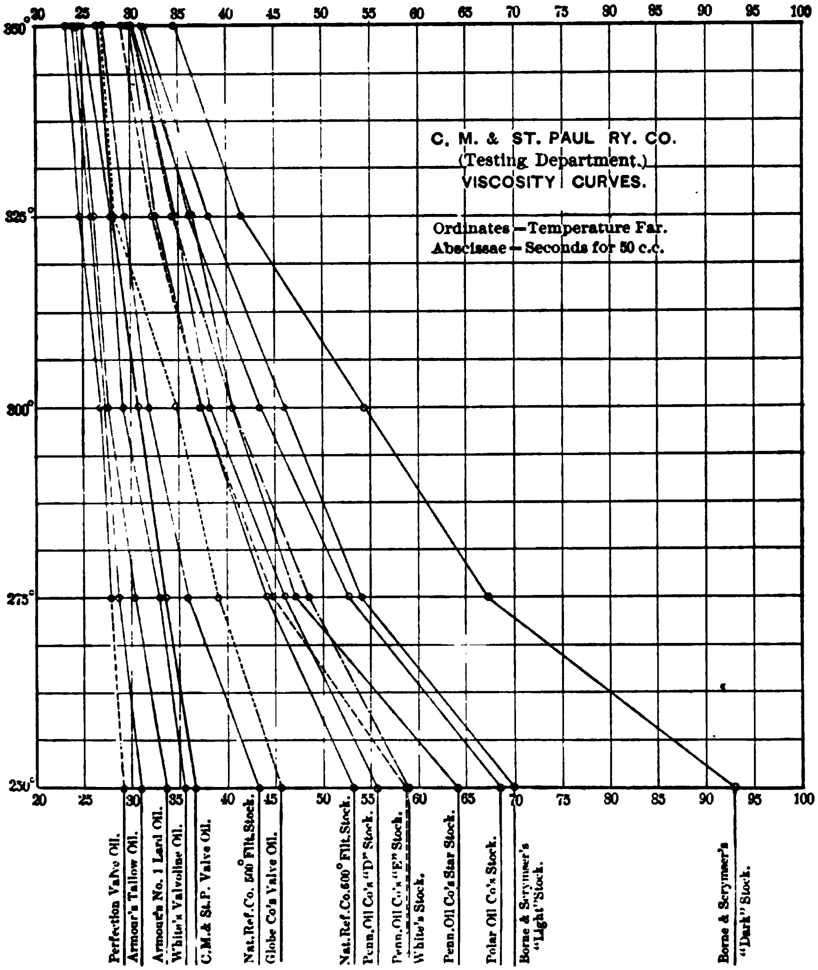


Fig 125.

A viscosimeter on an entirely different principle than the others already described is the Perkins instrument (G. H. Perkins, Supt. Atlantic Oil Refinery, Phila., Pa.) It consists of a cylindrical vessel of glass, surrounded by a proper heating vessel, and fitted with a piston. This piston fits into the cylinder to within $\frac{1}{1000}$ of an inch.

In practice, the cylinder is filled nearly full with the oil to be tested and the piston inserted. The time required for the piston to sink a certain distance into the oil is taken as the measure of viscosity. A full description of the apparatus will be found in *Transactions of the American Society of Mechanical Engineers*, **9**, p. 375.

J. Lew[1], introduces an instrument not only for the viscosity but also to include the internal friction of an oil. By these means it is claimed the lubricating value of the oil is absolutely determined.

The author states that the internal frictional resistances are different, and vary in the different oils at various temperatures. Formulas and methods are given by which coefficients are determined and used in the examination of the lubricating value of oils.

Figure 126 represents the viscosimeter designed and used in the chemical laboratory at the Stevens Institute of Technology.

It consists of a copper bath, *B*, surrounding the vessel, *A*, also of copper, and which holds the oil whose viscosity is to be determined. The tube *f* is of copper, but at *e* it is joined to a glass tube, which is extended to *d*—this latter is used for measuring the oil, and is carefully graduated. Sizes and dimensions of the apparatus are given in the figure.

This apparatus was designed to overcome two difficulties usually occurring in the use of other viscosimeters; *viz.*: *First*, loss of heat in the oil during its passage from the containing vessel to the receiving flask; and *second*, to have the chamber, *A*, of size to work small quantities of oil. *First.*—When the viscosity of an oil is taken at the ordinary temperature the measurement of the oil in the receiving flask will correctly indicate the amount of oil delivered through the aperture. The conditions are altered, however, when high temperatures are required, since the oil in running in a fine stream through the orifice is chilled in contact with the air, and if its temperature be taken

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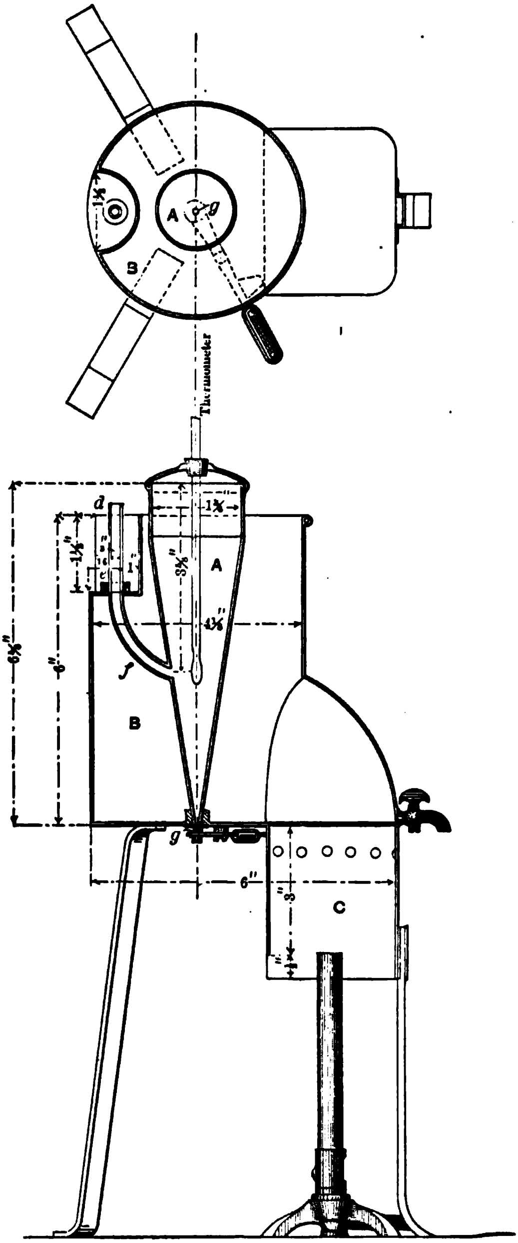
[1] *Ding. poly. J.*, 1891, 280.



AM.BK.NOTE CO.N.Y.

Fig. 126.

at the moment its volume is read in the receiving flask, a notable difference is indicated, depending upon the temperature of the room and of the oil before delivery.

In this instrument provision is made for reading the volume of the oil directly in the chamber *A* without any graduated receiving flask, as follows:

The tube *f e d* is graduated so that when the oil in the vessel *A* is at the proper level, the oil also reaches the upper graduated mark upon the tube *d e*. The lower graduated mark upon the tube indicates when twenty-five cc. of the oil have been delivered from *A* through the orifice *g*.

This graduation is absolutely correct for the purpose, and shows accurately the viscosity of the oil at any temperature, as indicated by the thermometer in *A*.

None of the oil in tube from *e* to *d* passes into *A* during the delivery of the twenty-five cc. through *g*, since the tube *f e d* is only partially emptied of its oil, the level of the oil in *A* after the deliveryof the twenty-five cc. still remaining above the point where the tube *f* enters *A*.

Second.—Oftentimes the samples of oil sent for examination do not exceed 100 cc. in bulk, an amount entirely too small if other tests are to be included.

Many forms of viscosimeters require 100 cc. of oil for the viscosity test, and not a few fifty cc.

I have found twenty-five cc. to be ample, provided the aperture at *g* is small enough to prevent a too rapid delivery of the oil and consequently render close readings and comparisons difficult. By making this orifice three sixty-fourths inch, sufficient time is secured to obtain accurate results.

If the operator prefers not to use the graduated tube *f e d* to measure the oil, a receiving flask, properly marked, can be placed under *g*, as in other forms of viscosimeters.

The plan suggested by Schubler that viscosities should be comparable with water is the only proper one and in the following determinations of viscosity the comparison is included:

	Seconds at 20° C.= 68° F.	Seconds at 50° C = 122° F.	Seconds at 100° C. =212° F.	Seconds at 150° C. = 302° F.	Seconds at 200° C. =392° F.
Water	51	......	......	......	
Prime lard oil	55	29	19	16	
No. 1 " "	70	30	18	16	
XXX " "	73	31	18	16	
Prime neat's foot oil	60	28	18	16	
White " " "	70	28	18	16	
Pure hoof oil	72	30	19	16	
Oleo oil	200	30	19	16	
Horse oil	64	35	17	16	
Gelatine oil	solid.	solid.	solid.	360	35
Rosin oil, 1st run	......	......	19	15	
" " 2d "	70	23	15	14	
" " 3d "	75	22	15	14	
Dog-fish oil (Pacific)	50	26	17	16	
Right whale oil (Pacific)	58	27	18	15	15
Natural bow head oil (Pacific)	47	27	18	16	
Bleached whale oil	52	27	18	16	
Natural winter Sperm oil (Pacific)	33	22	16	15	
Bleached sperm oil (Pacific)	29	22	16	15	
Natural spring sperm oil	30	22	17	16	15
Bleached spring sperm oil	32	22	16	15	
Natural winter whale oil (Atlantic)	53	26	17	16	
Bleached winter whale oil (Atlantic)	43	26	18	15	
Extra bleached winter whale oil (Atlantic)	55	28	18	16	
Natural spring whale oil (Atlantic)	57	26	18	16	
Bleached spring whale oil	52	26	18	16	
Porpoise head oil	34	30	16	15	
Sea elephant oil	51	36	17	16	
Bank oil	39	30	17	16	
Prime crude menhaden oil	39	24	17	16	
Brown strained " "	42	24	18	16	
Light " " "	40	24	17	16	
Natural winter menhaden oil	41	25	17	16	
Bleached " " "	34	24	17	16	
Extra bleached winter white menhaden oil	39	24	17	16	
Castor oil	730	95	23	17	15
"White seal" castor blown oil	......	185	28	20	20
Prime quality summer white cotton-seed oil	51	26	17	15	
Prime quality winter white cotton-seed oil	57	27	18	15	
Herring oil (Pacific)	56	26	18	16	
Rape oil	71	26	20	16	15
Olive oil	63	24	18	16	15

A chart of a few of the above oils is shown on the following page.

VISCOSITY TESTS.

a—Prime lard oil.	*i*—Castor oil.
b—Prime neat's foot oil.	*j*—"White seal" castor-blown oil.
d—Rosin oil (second run).	*m*—Prime qual. summer white cotton-seed oil.
e—Bleached whale oil (Pacific).	*n*—Rape oil.
f—Sperm oil (Pacific).	*o*—Olive oil.
g—Natural winter whale oil (Atlantic).	*p*—"Degras" oil.
h—Porpoise head oil.	*r*—Rosin oil (first run).

s—Gelatine oil.

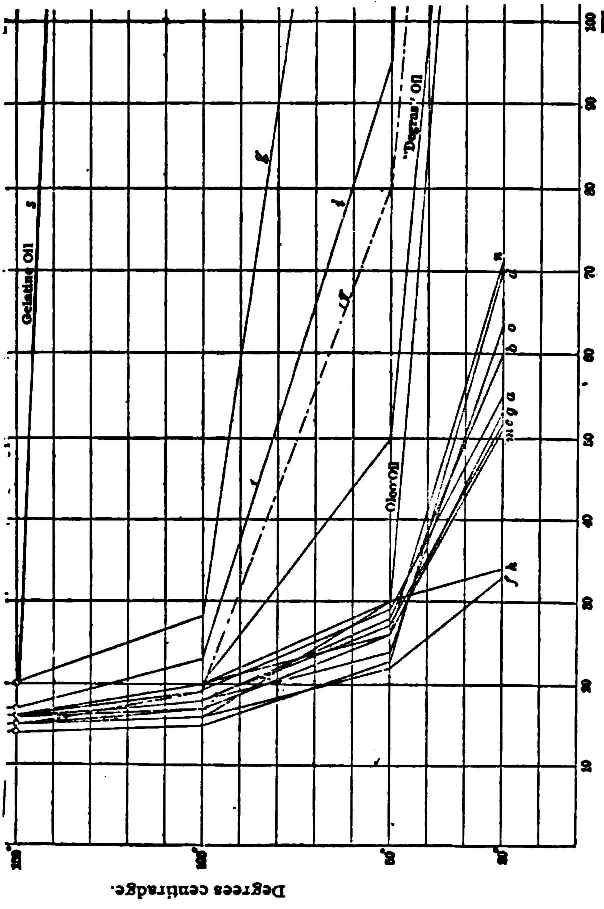


Fig. 127.

An examination of these tables and curves brings prominently forward the following facts :

That at high temperatures the variation in the viscosity of simple oils is very slight.

That "blown" oils, and "gelatine" oils, which are manufactured especially to give "body" to compounded oils fail in their purpose at high temperatures.

This is shown especially in Fig. 127, by the curves of the compounded oil, for instance, which at 20° C. remains solid, likewise at 50° C. and 100° C., but at 150° C. (302° F.) it indicates a viscosity of 360 seconds, and at 200 C., a viscosity of 35 seconds.

This "gelatine" oil is generally a compound of aluminum oleate, lard and petroleum.

Castor oil shows the highest variation of any of the simple oils, while sperm oil shows the least, and it is probably this property of the latter that has given it the reputation as the standard oil in lubrication.

Of the animal oils, lard oil ranks first in lubrication, followed in order by neat's foot, horse oil and tallow oil.

Generally speaking, the marine oils are the better lubricants, with the exception that acidity often rapidly forms in them, and so renders them valueless for the lubrication of many forms of machinery. The order of their value would be sperm oil, porpoise head oil, bleached menhaden oil, whale oil, dog fish oil, sea elephant oil and herring oil. Of the vegetable oils, rape oil is the recognized standard in lubrication. Its use for this purpose is very limited in this country, though in Germany and Russia large amounts are annually consumed.

Olive oil, while a good lubricant, is too high in price and its place has been taken in later years by refined cotton-seed oil. This latter oil, while seldom used alone in lubrication, is added to lard oil in proportions varying from twenty to fifty per cent., producing a mixture that lubricates nearly as well as pure lard oil, though acidity more rapidly develops than in lard oil alone. Castor oil is largely added to other oils to give high viscosity at ordinary temperatures, and to produce "body," which it loses at high temperatures. Its use for this purpose still continues in England, while in this country its application is limited.

The so-called " seal castors" and " blown oils" are made from cotton-seed oil, and are used in place of " gelatine" oil to produce high viscosity, at a much lower cost than " gelatine" oil.

" The Doolittle torsion viscosimeter"[1] recently introduced, (1893) is used in the railroad laboratories of the Philadelphia and Reading Railroad Co. It is briefly described as follows :

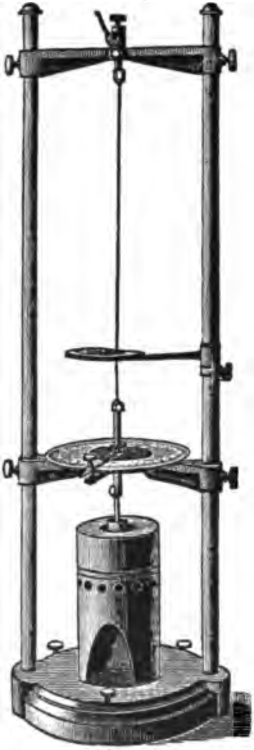


Fig. 128.

A steel wire is suspended from a firm support and fastened to a stem which passes through a graduated horizontal disk, thus measuring accurately the torsion of the wire. The disk is adjusted so that the index point reads exactly zero, thus showing that there is no torsion in the wire.

A cylinder two inches long by one and a half inches in diameter, having a slender stem by which to suspend it, is then immersed in the oil and fastened by a thumbscrew on the lower part of the stem to the disk. The oil is surrounded by a bath of water or paraffin wax according to the temperature at which it is desired to take the viscosity. This temperature being obtained while the disk is resting on its supports, the wire is twisted 360° by means of the knob at the top. The disk being released, the cylinder rotates in the oil by virtue of the torsion of the wire.

The action now observed is identical with that of the pendulum.

If there was no resistance to be overcome, the disk would revolve back to zero, and the momentum thus acquired would carry it to 360° in the opposite direction. What we find is that the resistance of the oil to the rotation of the cylinder causes the revolution to fall short of 360°, and that the greater the viscosity of the oil the greater will be the resistance and hence the retarda-

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[1] *J. Am. Chem. Soc.*, 15, 173.

tion. We find this retardation to be a very delicate measure of the viscosity of an oil.

There are a number of ways in which this viscosity may be expressed, but the simplest is found to be directly in the number of degrees of retardation between the first and second complete arcs covered by the pendulum. For example, suppose we twist the wire 360° and release the disk so that rotation begins. In order to obtain an absolute reading to start from, which shall be independent of any slight error in adjustment, we ignore the fact that we have started from 360°, and take as our first reading the end of the first swing. Suppose our readings are as follows:

Right, 350; left, 338; right, 328, and keeping in mind the vibrations of the simple pendulum we will see at once that we have read two complete arcs whose difference is 22° computed as follows:

1st arc, Right 350° + Left $338^\circ = 688^\circ$
2d arc, Left 338° + Right $328^\circ = 666^\circ$

22° retardation

In order to secure freedom from error we take two tests—one by rotating the wire to the right, and the second to the left. If the instrument is in exact adjustment these two results will be the same, but if it is slightly out, the mean of the two readings will be the correct reading.

It will also be noticed that if the exact retardation due to the oil alone is to be obtained we must subtract the factor for the resistance due to the air and the wire itself. These are readily obtained by allowing the cylinder to rotate in the air and determining the retardation exactly as we have done above. This factor remains constant for each instrument and is simply deducted from all results obtained.

Iodine Absorption.

The determination of the iodine absorption of an oil is probably the most important chemical test for recognition quantitatively in a mixture of animal or vegetable with mineral oils. Introduced by Hubl[1] it has since maintained this position,

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[1] *Ding. poly. J.*, 253, 281.

though other chemists have introduced the bromine absorption and others of similar character. They have not been adopted with the confidence of the iodine process.

Warren[1] draws attention to the fact that Chateau in his *Essais Personnelles*, p. 70, used the iodine absorption in a manner similar to Hubl many years previously.

In a mixture of two fatty oils with a mineral oil, the best results are obtained by saponifying and separating the fatty acids from the mineral oil. The iodine absorption of the mixed fatty acids is then taken, and where the nature of them has already been shown by color tests, etc., their proportion can be indicated by the following formula:

$$x = \frac{100\,(I-n)}{m-n}.$$

Where x = the percentage of one fat,
y = " " " the other,
I = iodine degree of mixture,
m = " " " fat x;
n = " " " " y.

The method is as follows:[2]

Twenty-five grams of iodine and thirty grams of mercuric chloride are each dissolved in 500 cc. of ninety-five per cent. alcohol, uniting the two solutions, and allowing to stand several hours before use.

It is then standardized by tenth normal thiosulphate solution. The process of the determination of the iodine absorption of an oil is as follows: One-tenth to five-tenths gram of the fat or oil is dissolved in ten cc. of purest chloroform in a well stoppered flask, and twenty cc. of the iodine solution added. The amount must be finally regulated so that after not less than two hours digestion the mixture possesses a dark brown tint; under any circumstances it is necessary to have a considerable excess of iodine (at least double the amount absorbed ought to be present), and the digestion should be from six to eight hours. Some potassium iodide solution is then added, and the whole diluted with 150 cc. of water, and tenth normal thiosulphate delivered

[1] *Chem. News*, 26. 188.

[2] Sutton: Volumetric Analysis, 343.

in till the color is nearly discharged. Starch is then added, and the titration finished in the usual way.

If more than two fatty oils are present in a mixture with mineral oil, the method of Warren[1] can be used.

The following determinations of the iodine absorption made in my laboratory are indicative of the variations of the absorption by the different oils:

Prime lard oil	76.4	77.2
No. 1 " "	69.8	69.9
XXX " "	65.1	65.6
Oleo oil	51.6	51.6
Prime neat's foot oil	80.1	82.0
Horse oil	82.3	82.5
Natural bow-head whale oil	130.5	131.1
" winter " "	121.1	126.0
Extra bleached winter white oil	124.9	126.1
Bleached spring " "	126.1	126.2
Crude sperm oil	82.3	82.3
Prime quality winter white cotton-seed oil	114.2	114.9
" " summer " " " "	110.2	110.6
" " winter yellow " " "	115.9	118.6
" " summer " " " "	104.0	104.4
Olive oil	81.0	83.0
Herring oil	122.1	123.8
Dog-fish oil	102.7	104.7
Porpoise head oil	28.9	29.1
Rosin oil, second run	92.1	93.4
" " third "	90.4	92.2

Flash and Fire Test.

The flash point is the degree of temperature at which ignitable volatile vapors are given off by the oil, producing a flash when brought in contact with a small flame. The fire test is a continuation of the flash test until the oil permanently ignites. A simple apparatus that gives approximate results is shown in Fig. 129. It consists of a porcelain crucible two and one-eighth inches wide at the top, five-eighths inch wide at the bottom and one and one-half inches deep. This is surrounded by an asbestos pad three and one-half by three and one-half inches and one-eighth inch thick. This prevents the direct contact of the gas flame upon any portion of the crucible except the base. The oil to be

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[1] *Chem. News*, 62, 215: *J. Anal. Appl. Chem.*, 5. 215.

tested is placed in the crucible, a thermometer inserted at such a depth that the bulb is just covered by the oil, and the heat applied. The rise of temperature in the oil should not exceed 2° F. per minute.

The "test-flame" (the smallest possible) is passed over and across the surface of the oil once every minute beginning at 100° F.

Oils that flash below 110° F. are considered unsafe for lighting purposes, and for lubricating purposes; oils should not flash under 250° F.

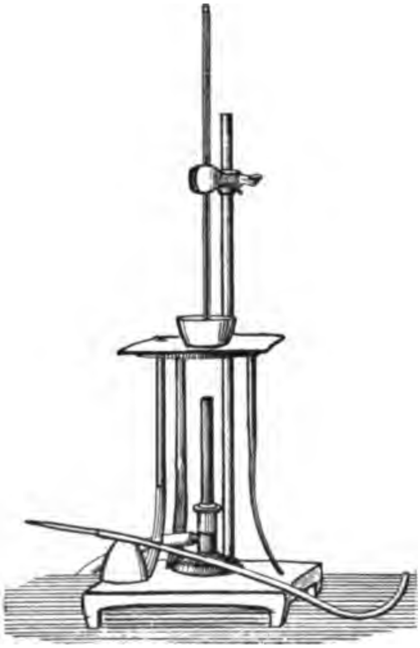

Fig. 129.

Fig. 130.

The Cleveland cup oil tester is very similar to this instrument in design and operation, with the exception that the porcelain crucible is replaced by a copper one of the same size and heated in a sand-bath instead of being surrounded by an asbestos pad.

Tagliabue's open tester, has a very extensive use in the oil trade. It consists (Fig. 130) of a copper cylinder, *B*, into which fits the copper water-bath, *A*, and a glass cup, *D*, which contains the oil to be tested. This apparatus has been superseded somewhat by another form of open tester. The "Saybolt" which is used by the chemists of the Standard Oil Co., and officially adopted by the New York Produce Exchange. It consists of a water-bath, *F*, (Fig. 131) surrounding an inner cup containing the oil. An induction coil, *E*, furnishes an induction spark that passes over the oil. Batteries for generating the current are situated under the frame, *C*.

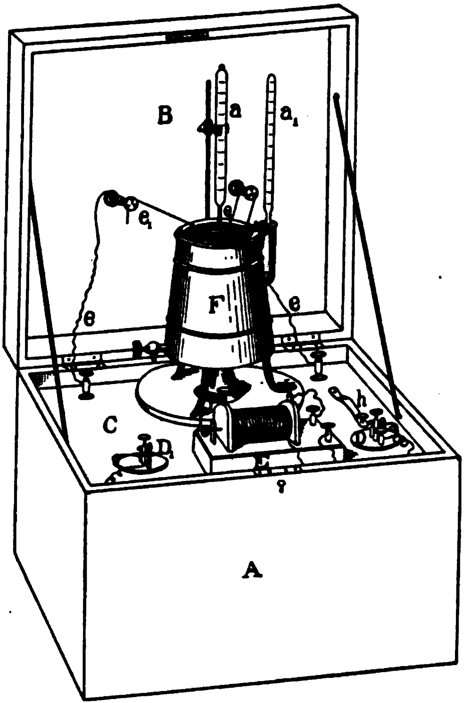

Fig. 131.

All open cup testers give higher readings for the flash test than closed testers and it is generally conceded that the closed testers admit of more accurate determinations.

The Abel closed tester Figs. 132, 133, has been adopted by the English government, and in a modified form (Pensky-Martens) by the German government as the official instrument for this purpose.

The specifications for this instrument require that the oil cup be a cylindrical vessel, two inches in diameter, two and two-tenths high (internal), with outward projecting rim five-tenths inch wide, three-eighths inch from the top, and one and seven-eighths inches from the bottom of the cup. It is made of gun-metal or brass (17 B. W. G.) tinned inside. A bracket, consisting of a short stout piece of wire, bent upward, and terminating in a point, is fixed to the inside of the cup to serve as a gauge. The distance of the point from the bottom of the cup is one and a half inches. The cup is provided with a close-fitting, overlapping cover, made of brass (22 B. W. G.) which carries the thermometer and test-lamp. The latter is suspended from two supports from the side by means of trunnions, upon which it may be made to oscillate; it is provided with a spout, the mouth of which is one-sixteenth of an inch in diameter. The socket which is to hold the thermometer is fixed at such an angle, and its length is so adjusted, that the bulb of the thermometer, when inserted to full depth, shall be one and a half inches below the center of the lid. The cover is provided with three square holes, one in the center, five-tenths inch by four-tenths inch, and two smaller ones, three-tenths inch by two-tenths inch, close to the sides and opposite to each other. These three holes may be closed and uncovered by means of a slide moving in groves and having perforations corresponding to those on the lid. In moving the slide so as to uncover the holes, the oscillating lamp is caught by a pin fixed in the slide and tilted in such a way as to bring the end of the spout just below the surface of

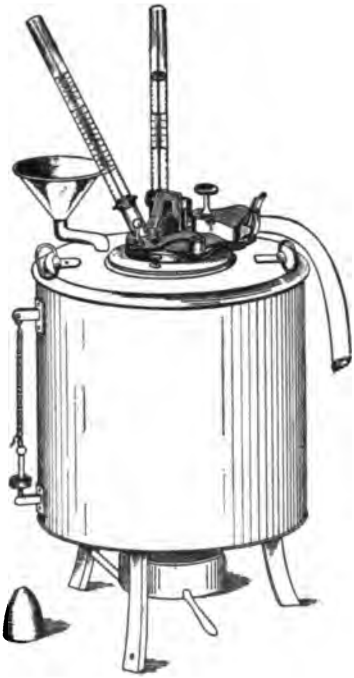
Fig. 132.

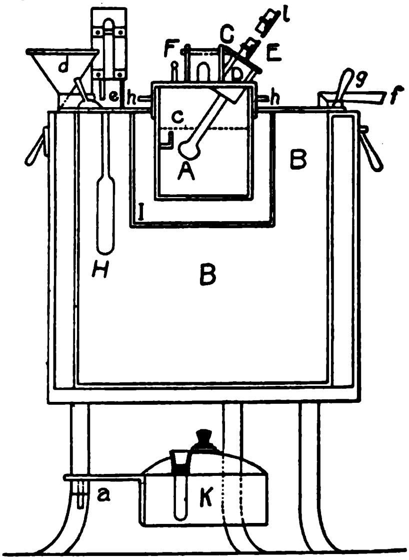

Fig. 133.

the lid. Upon the slide being pushed back so as to cover the holes, the lamp returns to its original position.

The flash test of this apparatus is about 27° F. lower than the open cup apparatus, so that 73° F. Abel test is equivalent to 100° F. test, open-cup test.

The Pensky-Martens closed tester, Figs. 134, 135, in action is very similar to the Abel closed tester. The apparatus of Treumann, Figs. 136, 137 is used by the chemists of the Prussian railways for the determination of the flash and fire test of both illuminating and lubricating oils.

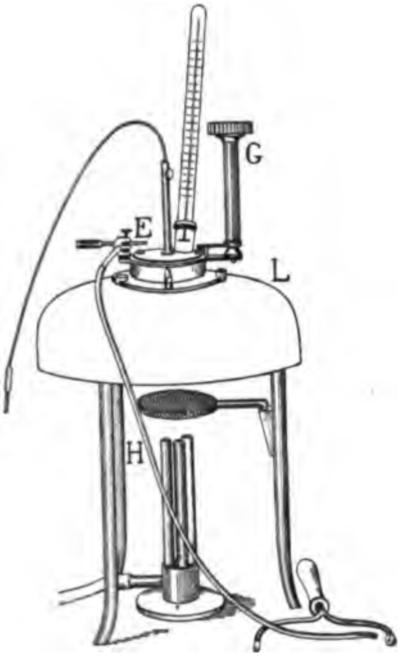

Fig. 134.

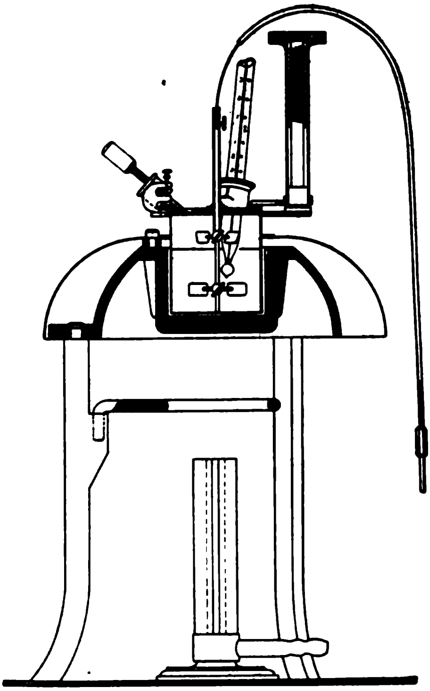

Fig 135.

It is very similar in construction and operation to the Cleveland cup, in use in this country for the same purpose, with the exception that the oil is placed in a porcelain crucible, *a*, Fig. 137, instead of a copper one as in the Cleveland cup.

The larger containing vessel is of iron and contains sufficient sand to raise the bottom of the crucible containing the oil, one-half inch from the point of contact of the flame.

The flash and fire tests are required of all lubricating oils as a test of their power to resist combustion by overheating in work. Valve oils with mineral stock are especially liable to have low flash points caused by imperfect distillation in their manufacture. They should be free from any of the lighter oils (naphtha, keroseñe, etc.,) and should not flash under 300° F. For cylinder oils the requirement is much higher. Animal and vegetable oils used in lubrication rarely flash under 400° F.

Fig. 136.

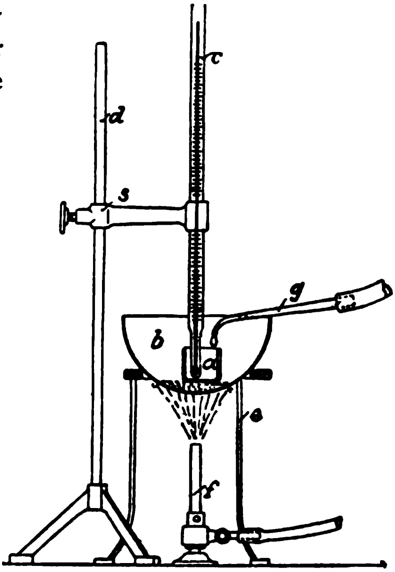

Fig. 137.

Acidity.

Acidity in oils is generally due to a partial decomposition of the oil with liberation of fatty acids. These latter act as corrosive agents, attacking the metal bearings of machinery, forming "metallic soaps" and producing gumming and thickening of the lubricant.

Properly refined mineral oils are free from acidity, but nearly all animal and vegetables oils possess it more or less.

In palm oil, for instance, the free fatty acids vary from twelve to eighty per cent. In eighty-nine samples of olive oil intended for lubricating purposes, D. Archbutt[1] found from 2.2 to 25.1 per cent. of free acid (oleic) the mean being 8.05 per cent.

Oleic acid cannot be present as a constituent of a pure mineral oil; still the acid test should be made, since poorly refined mineral oils are liable to contain small amounts of sulphuric acid left in the process of refining. The sulphuric acid is easily indicated by warming some of the oil with distilled water, adding a few drops of hydrochloric acid (dilute) and solution of barium chloride. A white cloud or precipitate shows the presence of sulphuric acid.

The action of free acid on journals, bearing, etc., as a corrosive element, has led many engineers to include a test of free acid direct upon copper and iron.

This is done by suspending weighed pieces of sheet copper and iron in the different oils, for a number of days, heating if necessary, and determining the amount of metal dissolved by the oils.

While this test may be indicative of the acidity of oils, no ratio exists between the action upon copper and iron or even between the oils themselves in this respect, owing to the varying quantity of acid in the same oils.

The results of a few tests are shown in the following table:

Name of oil.	Copper dissolved after 10 days.	Iron dissolved after 24 days.
Linseed oil	0.3000 gram.	0.0050 gram.
Olive oil	0.2200 "	0.0062 "
Neat's foot oil	0.1100 "	0.0875 "
Sperm oil	0.0030 "	0.0460 "
Paraffin oil	0.0015 "	0.0045 "
Lard oil	....	0.0250 "

The following is the method for determining the acidity of oils, as used in many of the railroad laboratories:

Materials Required.

One fifty cc. burette, graduated to tenths.

Two ounces alcoholic solution phenolphthalein.

[1] *Analyst*, 9, 171.

Three ten cc. pipettes.

One druggist's graduate, four ounces.

One gallon ninety-five per cent. alcohol.

One dozen four ounce sample bottles.

One thermometer graduated from 15° to 215° F., and bearing the certificate of the Yale Thermometer Bureau.

Two hydrometers 15° to 25° and 25° to 35° B., each degree graduated to tenths (Tagliabue's.)

One hydrometer jar.

One quart caustic potash solution of such strength that 31.5 cc. exactly neutralize five cc. of a normal solution of sulphuric acid (contains forty-nine mgms. per cubic centimeter of sulphuric acid.)[1]

Take two ounces of alcohol and warm tq about 100° F.; add ten drops of alcoholic solution of phenolphthalein. Fill the burette to the top of the graduation with the caustic potash solution; then add solution drop by drop to the alcohol until it assumes a pink tint. Add ten cc. of the oil to the alcohol, refill the burette with the potash solution and add the latter until the mixture of oil and alcohol maintains a pink color after thorough shaking. Read off the number of cc. of potash solution used, and this amount divided by two, gives the per cent. of free acid. For example, if 10.6 cc. caustic potash solution have been used, the oil contains five and three-tenths per cent. of free fatty acid.

Lard and tallow are very liable to have considerable amounts of free acid. The specification of purchase therefore generally states the limits of free acid permitted.

Maumené's Test.

The rise of temperature produced when sulphuric acid is brought in contact with certain oils was first investigated by Maumene, and the results of his experiments published in *Comptes Rendus*, 35, 572.

The subject has been investigated by Fehling, Faist, L. Archbutt, C. J. Ellis, A. H. Allen and others, with the result that

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[1] Hydrometers and thermometers should be procured through Chas. A. Tagliabue, New York.

this test has been generally accepted as of importance in the distinction of oils in mixtures.

When a mixture of oils has been analyzed and the components recognized the proportions oftentimes can be determined by this reaction; that is to say, suppose the oil under examination to show a rise of temperature of 80° C., and the oils found by analysis to be lard oil and menhaden oil; their relative proportions can be determined by the following formula:

$$W_1 = W_3\frac{t_3 - t_2}{t_1 - t_2}.$$

$$W_2 = W_3\frac{t_3 - t_1}{t_2 - t_1}.$$

W_1 = proportion by weight of menhaden oil.
W_2 = " " " " lard "
W_3 = weight of mixture.
t_1 = temperature of menhaden oil.
t_2 = " " lard "
t_3 = " " mixture.

The method is as follows:

Fifty grams of the oil are placed in a narrow tall beaker and ten cc. of C. P. sulphuric acid added drop by drop with stirring and the rise of temperature during the operation noted.

Lard oil alone when treated with sulphuric acid gives a rise of temperature of 40° C.; menhaden oil, under similar conditions, a rise of 128° C. Using these values in the above formula we obtain 54.6 per cent. lard oil and 45.4 per cent. menhaden oil.

In the mixture containing a mineral oil mixed with animal, marine or vegetable oil the distinction would be even more pronounced, since the mineral oil shows but a very slight increase of temperature (generally from 2° C. to 5° C.). The increment of temperature would be dependent upon the other oil added to the mineral oil.

Briefly stated, the rise of temperature of the following oils would be:

	Name of Observer.				
	Maumene. °C.	Schaedler. °C.	Archbutt. °C.	Allen. °C.	Stillman. °C.
Lard oil	40			41	39.5
Tallow oil	41–43				39
Neat's foot oil	45	50	43		40
Oleo oil			37½	38½	37
Elain oil					38
Sperm oil			51	45–47	48
Whale oil			92	91	92
Menhaden oil			123–128	126	128
Dog-fish oil					80
Cod liver oil	102–103	103		113	110
Crude cotton-seed oil		69.5	70	67–69	74
Rape oil	58				60
Castor oil	47	48	46	65	45
Olive oil	42	43	41–45	41–43	42
Rosin oil		28		18–22	10
Mineral lubricating oil				3–4	3
Earth nut	67	67	47–60		
Rosin oil		28		22	10
Sea elephant					65

Attention is drawn to the differences in the determinations in rosin oil.

Rosin oil of the first run is a white, opaque, thick liquid containing all of the water of the rosin from which it is distilled, and it is this water that causes the rise of temperature above 10° when the oil is mixed with the sulphuric acid.

Rosin oils of the second and third runs are clear, limpid, dark red colored fluids, practically free from water, and when treated with sulphuric acid do not indicate more than 10° rise of temperature.

From these tests it is concluded that both Schaedler and Allen tested rosin oil that was a mixture of the first and second runs, or of an oil not properly separated into the different distillates.

Color Reactions of Oils with Nitric and Sulphuric Acid.

Of the many color tests introduced for the identification of simple oils, preference is given to Heidenreich's sulphuric acid test and Massie's nitric acid test.

The color reactions of Chateau[1] in which barium poly-sulphide

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[1] Spon's Encyclopedia, 4, 1472–1475.

zinc chloride, stannic chloride, phosphoric acid and mercuric nitrate, in solutions, are used, while very interesting, seldom are of any advantage over the two tests noted above. Glassner's[1] nitric acid reactions are practically the same in results as Massie's so that no advantage would be obtained in including the former.

Heidenreich's test is as follows:

A clear glass plate is placed over a piece of white paper, ten drops of the oil under examination are placed thereon, and one drop of concentrated sulphuric acid is added.

The color produced when the acid comes in contact with the oil is noticed as well as the color produced when the two are stirred with a glass rod. Many oils give off characteristic odors during the reaction, especially neat's foot oil, whale oil and menhaden oil.

Massie's test is thus performed:

Nitric acid of specific gravity 1.40, free from nitrous acid, is mixed in a test tube with one-third its volume of the oil, and the whole agitated for two minutes.

The color of the oil after separation from the acid is the indication.

In mixtures of oils, the characteristic colors produced, by either Heidenreich's or Massie's test, are often clouded, and in many instances no inferences can be drawn, yet with single oils the reactions are often distinctive and sufficiently strong to give confirmatory results.

In cod liver oil, or whale oil, when mixed with a mineral or even vegetable oil, the characteristic brilliant violent color produced with sulphuric acid cannot be mistaken. This color, due to the presence of cholic acid, is found in most of the fish oils, but is much more pronounced in cod liver oil.

The following table will indicate the colors produced by Heidenreich's and Massie's test.

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[1] *Chem. Centrbl.*, *1873*, 57.

	Heidenreich's test.		Massie's test.
	Before stirring.	After stirring.	
Lard oil	Yellow.	Brown.	Yellow.
Tallow oil	Yellow.	Orange.	Colorless.
Neat's foot oil	Yellowish.	Red brown.	Red.
Oleo oil	Colorless.	Orange.	Pink.
Elain oil	Light green (turning to brown).	Brown.	Orange red.
Sperm oil	Brown with purple streaks.	Reddish brown.	Red.
Whale oil	Red violet.	Violet brown.	Red.
Menhaden oil	Red.	Brown.	Dark red.
Dog-fish oil	Violet.	Dark brown.	Orange.
Cod liver oil	Red violet.	Dark brown.	Orange red.
Crude cotton-seed	Brilliant red.	Brown.	Brown.
Ref'd cotton-seed	Reddish brown.	Red.	Orange red.
Rape oil	Yellow brown.	Brown.	Orange.
Castor oil	Lgt. yel. to brown.	Pale brown.	Orange.
Olive oil	Light green.	Greenish to light brown.	Yellow to greenish.
Rosin oil	Brown.	Brown.	Orange.
Earth nut oil	Yellow to orange.	Greenish.	Reddish.

The oils made use of in lubrication can be separated into two groups: saponifiable and unsaponifiable. To the former belong all the fatty oils; to the latter the mineral and rosin oils.

The method of Lux[1] is made use of to determine if any fatty oils are present in a mineral oil.

If rosin oil is suspected to have been added to the mineral, it can be identified by the method of Holde[2] or the process of E. Valenta[3] can be used.

These three tests will indicate, qualitatively, the presence of any fatty or rosin oil in a mineral oil. It is rarely, in the better class of lubricating oils, that more than one oil is added to a mineral oil, such, for instance, as lard oil, or tallow, in which case saponification easily separates the two oils, and identification of each by special tests can then be made.

When, however, the oil added to the mineral oil itself contains an adulterant, such as lard oil to which cotton-seed oil has been added, then the fatty acids separated by saponification will require a more extended examination to prove the presence of both lard oil and cotton-seed oil.

[1] *Ztschr. Anal. Chem.*, 24, 347.

[2] Mittheil der Konig. tech. Versuchsanstalten, *1890*, 19.

[3] *Ztschr. anal. Chem.*, 25, 441.

The following skeleton scheme is given to show the application of the above upon a lubricating oil that qualitative analysis has shown to contain mineral oil, lard oil, and cotton-seed oil.

Twenty grams of the oil are weighed out in a No. 3 beaker, 100 cc. of an alcoholic solution of potash (eighty grams potassium hydroxide to one liter alcohol of ninety-eight per cent.) are added, and heat applied with stirring until the alcohol is all driven off; add 100 cc. water, heat with agitation, cool, add fifty cc. ether, transfer to separatory funnel, stopper, shake well and allow to stand two hours. Draw off the soap solution.

1. *Soap solution* (containing the fatty acids of the lard and cotton-seed oils). Heat ten minutes nearly to boiling, cool, acidify with dilute sulphuric acid, allow to stand a few hours; collect the separated fatty acids; determine their weight, then test as follows: **First portion: Determine the "melting-point."** **Second portion: Determine the "iodine absorption" and their rates by formula:** $x=100\,(I-n)$ $x=m-n.$	**2. *Ether solution* remaining in the separatory funnel is transferred to a flask, the ether distilled and the mineral oils weighed.**

There are several methods for the quantitative determination of the amounts of vegetable and animal oils when mixed with each other, or when the mixture is incorporated with a mineral oil. The determination of the iodine absorption is the most delicate and correct provided no fish blubber or olive oils are present.

If the fatty acids have been separated, by saponification, from a mineral oil, this iodine value can also be determined. Consult soap analysis, for table of constants.

The method of *Salkowski*[1] depends upon the fact that vegetable oils (except olive) contain phytosterol and that animal fats (butter excepted) are free from it, containing cholesterol, the latter not being present in vegetable oils.

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[1] Benedikt: Oils, Fats and Waxes, 255.

Fifty grams of the sample free from mineral oil are saponified with alcoholic potash ; the soap solution is diluted with a liter of water and exhausted with ether. When the two layers have separated, the aqueous layer is run off and the ethereal liquid filtered and evaporated to a small bulk. To insure complete absence of unsaponified fat, it is best to saponify again with alcoholic potash and to repeat the exhaustion with ether. The ethereal layer is then washed with water and the ether evaporated in a deep basin. The residue is next dissolved in hot alcohol, the solution boiled down to one or two cc. and the residue allowed to cool. If phytosterol or cholesterol be present, crystals will separate out. They are dried on unglazed porcelain and their melting points determined.

The saponification value of oils is often made use of for identification : but as this value varies with the age of the oil, it is extremely difficult to obtain concordant results, and as the majority of oils have a saponification value of 193, excepting rape-seed oil and castor oil which are lower, it can not be relied upon. It however is of value in determining the amount of liquid waxes in the presence of oils.

Wool-grease is used to some extent in the cheaper grades of lubricants, the consumption for this purpose increasing yearly. It is unsaponifiable and, if present, will be found in the ether extract with the mineral oil, in the analysis as usually conducted of a mixed lubricating oil.

Degras or *sod oil* is a waste product obtained in the chamoising process. It is largely derived from whale oil or poor quality of cod liver oil used in chamoising.

The English-German method of treating skins produces sod oil as a waste product. The French method produces Degras. These fats are largely used in the production of cheaper lubricants.

Consult Benedikt: Oils, Fats and Waxes, 589; *J. Am. Chem. Soc.*, (Bush), 16, 535.

Bone Fat is made use of in lubrication mixed with mineral oils. It is recovered from bones, either by boiling with water or extracting with solvents. It does not readily become rancid. Its examination is made similarly to that of tallow.

Coefficient of Friction.

The ratio of the force required to slide a body along a horizontal plane surface to the weight of the body is called the coefficient of friction. It is equivalent to the tangent of the *angle of repose*, which is the angle of inclination to the horizontal of an inclined plane on which the body will just overcome its tendency to slide. The angle is usually denoted by φ, and the coefficient by f.

$f = \tan \varphi$. (Kent.)

Of the various machines used for this purpose nearly all are deficient in conducting tests under extreme pressure. However as all the tests are relative, an idea of the value of a lubricant can be formed by a series of comparative tests upon the same instrument.

G. B. Heckel thus describes the Thurston and Henderson-Westhoven machines: The primary idea of determining durability is to determine how much rubbing a lubricant will withstand before exhaustion of its power to maintain the friction at some agreed minimum. For this there is no device superior to the Thurston oil-tester, in which a pair of brasses are forced against a journal in opposite directions by a spring being lodged in a pendulum which is free to swing about the journal, the friction being measured by the inclination to the vertical of a line join-

Fig. 138.

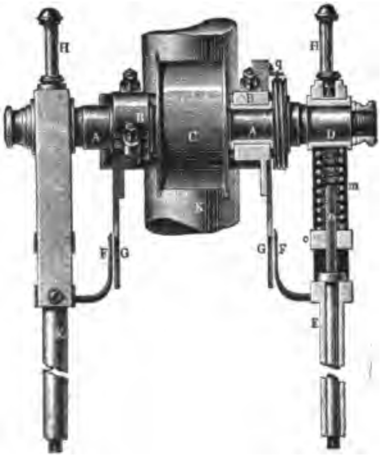

Fig. 139.

ing the center of the journal and the center of gravity of the pendulum. The defects of this machine lie in the infinitely variable rate of metallic wear between rubbing surfaces, which contaminates the oil before it has been exhausted, as well as in the escape of the lubricant between the surfaces.

These imperfections have been overcome in the Henderson machine or the so-called Henderson-Westhoven machine, a modified Thurston tester. (Figs. 138, 139.)

With this machine lubricants can be tested at the same moment for the degree of heat developed in the bearing surfaces as well as their friction reducing qualities.

The journal, *A*, rests upon the supporting beds, *BB*, and is revolved by the pully, *C*. This journal, *A*, extends on both sides beyond the supports, *BB*, and the projecting ends are embraced by brass boxes *DD*, to which are fastened the pendulum parts *EE*. Strong spiral springs *mm*, in the interior of the pendulum arms, force the lower pair of brasses, *DD*, against the journal, *A*, and the pressure of these springs may be regulated by means of the screw, *N*. A pointer attached to the movable block, *o*, indicates on the scale, *P*, as in a spring balance, the thrust of the spring against its bed, in kilograms per cubic centimeter. By the revolution of the journal, *A*, the swinging arms, *EE*, are actuated by friction in the direction of the motion, and the degree of their deviation from the vertical is read by means of the pointers, *FF*, on the quadrants *GG*. On many machines the scales give, besides the deviation, also the coefficient of friction which has been calculated from the former.

In the upper brasses, *DD*, a thermometer, *H*, is fixed to show the degree of heat developed by the friction, and the revolution counter, *J*, actuated through the endless screw, *q*, records the revolutions of the journal, *A*. The column, *K*, through its two arms, *L*, carrying the boxes, *BB*, serves to support the entire device.

In operation the oil to be tested is introduced by means of a small glass tube or pipette, through an orifice in the upper brasses, *DD*, the journal having been thoroughly cleaned. The position of the thermometer and of the revolution counter are noted, and the journal is then put into motion with 200 or 300 revolutions per minute. At each succeeding five-hundredth or thousandth revolution the temperature and the degree of deviation of the pendulum arms, as shown by the quadrant, are noted, and when the friction has raised the temperature in the boxes about 30° (usually in about half an hour) the machine is stopped. In figuring up results, the sample of oil which, with an equal rise in temperature at the point of friction, gives the slightest deviation of the swinging pendulum arm, and the greatest number of revolutions, is regarded as the best. The advantages noted in this device are its facilities for testing materials under any pressure, even up to the load limit on a freight car axle; the number of data obtainable at one time; and the ease with which two simultaneous tests of competing oils can be made on the one machine.

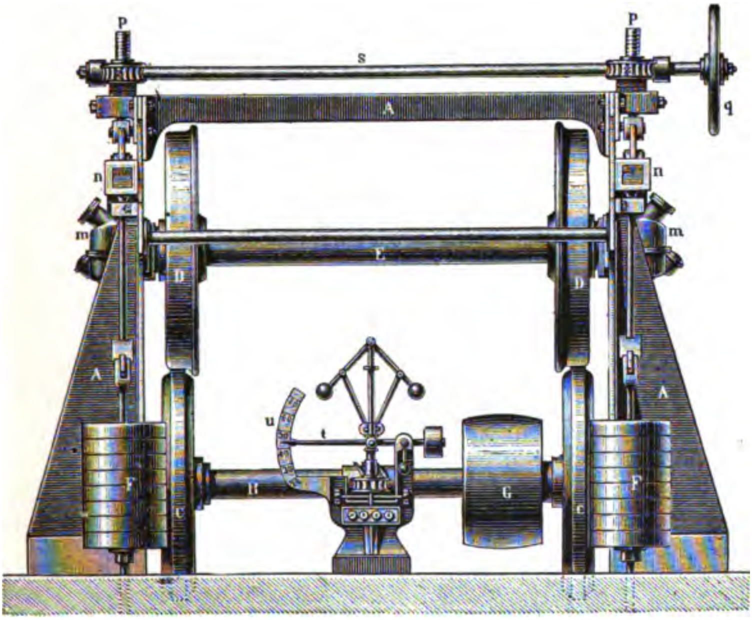

Fig. 140.

The apparatus used for testing lubricants by the officials of the Paris-Lyon Railway is shown in Figs. 140, 141. Here the conditions are maintained as nearly as possible as would occur in railroad practice, the friction being determined by means of two freight-car wheels.

The heavy cast-iron frame, *A*, stands upon a firm stone foundation and carries the shaft, *B*, on which are fastened the two friction wheels, *CC*. These are placed at the same gauge as the railroad track. Two ordinary car wheels, *DD*, with axle, *E*, are placed above and in contact as shown in the figure. The car axle, *E*, is fitted at each end into the axle boxes, *mm*. The boxes have the same arrangement as those in the railroad cars

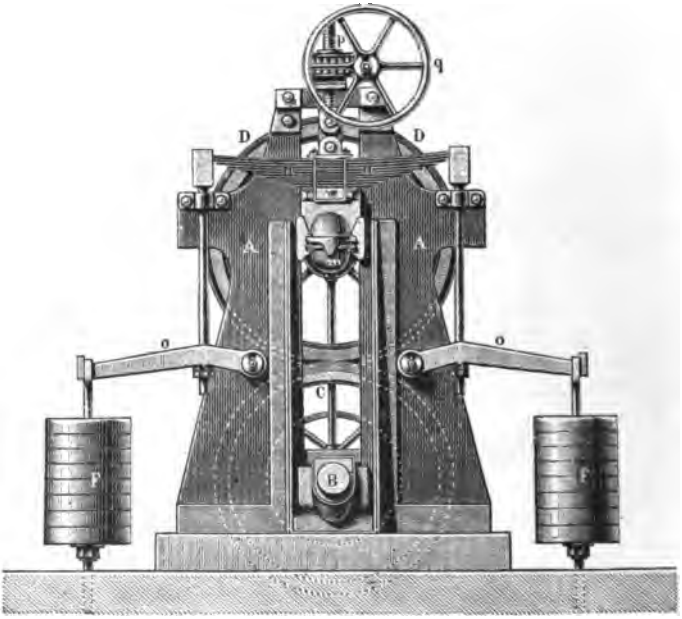

Fig. 141.

and serve for the reception of the lubricant. Resting on each side of the axle boxes are the strong springs, *nn*, Fig. 140, on the end of which the weights, *FF*, work by means of the levers, *oo*. By taking off or putting on of weights, *FF*, *E* can carry any load desirable.

On the lower shaft is the driving wheel, *G*, also a screw by which the movement of the shaft is carried to a figured dial. This dial sets not only the index showing the number of revolutions but also the index needle, *t*, in motion which indicates on the scale, *u*, the approximate rapidly of the wheel-rims in kilo-

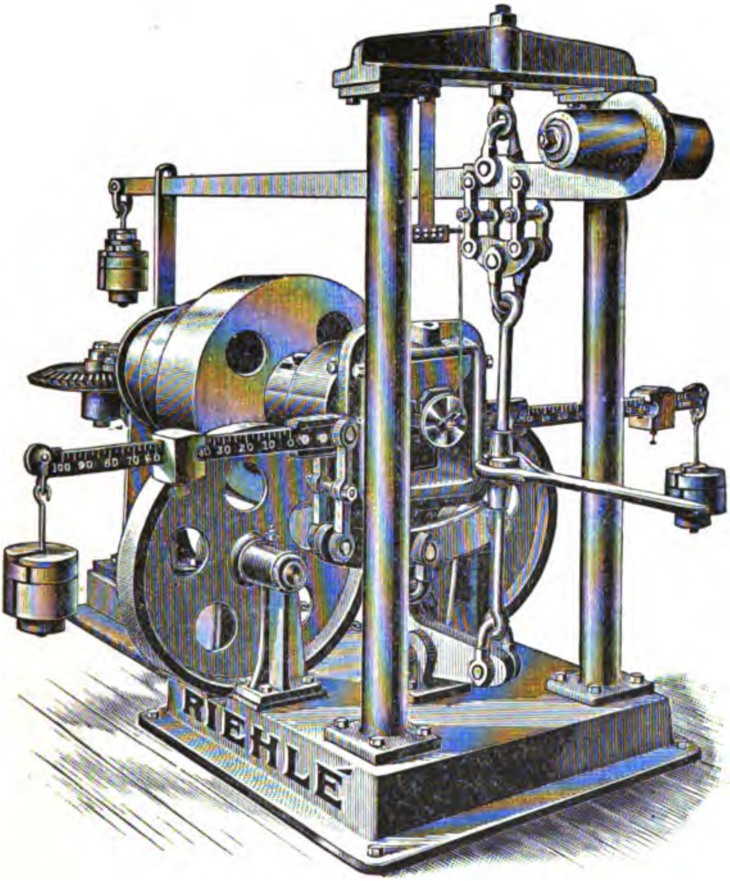

Fig. 142.

Extreme length	7½ feet.
Extreme height	6 feet.
Extreme width	6½ feet.
Weight	6250 pounds.
Shipping weight	6500 pounds.

meters per hour. The two friction wheels, *cc*, are turned eccentrically about two and five-tenths mm. that by the motion a weak vertical oscillation arises which is communicated to the upper wheels whereby the rattling of the wheels upon the car track is imitated.

In making a trial, the lubricant to be tested is placed in the thoroughly cleaned axle boxes, *mm*, the springs are lifted to the utmost release of the upper shaft and the lower shaft is placed in rotation. Not until the whole is in motion are the springs brought down, and later loaded with the intended weight. The oil which by this test carries the burden with the greatest rapidity without heating of the axle-boxes is to be considered the best. By this apparatus it is possible to judge of the practical working of an oil or compounded oil, and especially if the car axles would become heated, a point of vital importance as regards the use of the lubricant.

Another instrument of a similar design is the Riehlé, (Fig. 142,) in use in many railroad laboratories in the United States, for testing lubricants. The capacity is 20,000 pounds; it determines the coefficient of friction, the pressure per square inch of journal and records the temperature.

It consists of a Master Car Builder's Axle journal, which is removable from the main spindle. This journal is made to revolve by cone pully at different speeds, and in either direction, and can be loaded to different pressures per inch by means of the lever system. The oil can be supplied through a hole in the top, which is tapped to receive a sight-feed oiler, or funnel, or other arrangement.

The friction is weighed on the beams, which are arranged in double system to balance each other, allowing the machine to be run in either direction. The opening in the frame over the journal is made large enough to take a regular car box if desired.

The frame and beams can be raised by rope sling and hoist for change of journal, cleaning up, etc.

There is an end motion of about one-fourth to three-eighths inch given to the axle by the gearing shown at the end, giving a natural movement like cars. The weighed end of spindle runs loose on large rollers, to avoid friction and heating.

An oil tested upon the tester may show a fine lubricant, while put under practical working upon a freight car (for instance) would prove vastly inferior. This very often happens, and it has led many engineers to test each oil by a long run, with the particular kind of machinery upon which it is to be used.

A record-blank used by the engineers of the Baltimore and Ohio Railroad, for testing oils upon their locomotives is given herewith. It is a point in instance. After experimenting months upon an oil its work is established so that a practical comparison can be made with other brands of similar composition for the same purpose.

BALTIMORE AND OHIO RAILROAD.

Subject............................. 189

....

Engineer of Tests,

DEAR SIR: Below please find report from locomotives inspected this day.

Engine No.	Train No.	Service.	Engineer.	Fireman.	Left.	Time.	Arrived.	Time.	Miles run.	Average speed, miles per hour.	Condition of bearings.	Kind of oil used.		Miles run per pint of oil allowed		Miles run per pint of oil used.		Number of drops per minute.	
												Cylinder.	Journal.	Cylinder.	Journal.	Cylinder.	Journal.	Cylinder.	Journal.

........................*Inspector.*

BALTIMORE AND OHIO RAILROAD. OFFICE OF SUPERINTENDENT OF MOTIVE POWER.

(Specifications for Compound Oils.)

DETAIL SPECIFICATIONS.

Engine and Passenger Car Oil.

This oil must conform to the following requirements.

1. It must have a flashing point from October 1 to May 1, above 200° F.; from May 1 to October 1 the flashing point must be above 250° F.
2. From October 1 to May 1 it must have a cold test below 15° F.
3. It must show no sediment in fifteen minutes when five cc. are mixed with 100 cc. of gasoline of 85° B.
4. It must contain not less than thirty per cent. saponifiable animal oil.
5. Its gravity must be between 26° and 30° B.

Cylinder Oil.

This oil must conform to the following requirements:

1. It must have a flashing point above 440° F.
2. It must contain not less than thirty-five per cent. of saponifiable animal oil.
3. It must show not more than six per cent. of fat acid or its equivalent.
4. It must not show any precipitation when five cc. are mixed with 100 cc. of gasoline of 85° B.

Freight Car Oil.

This oil must conform to the following requirements:

1. It must have a flashing point from October 1 to May 1 above 200° F.; from May 1 to October 1 the flashing point must be above 250° F.
2. From October 1 to May 1 it must have a cold test below 15° F.
3. It must show no sediment in fifteen minutes when five cc. are mixed with 100 cc. of gasoline of 85° B.
4. It must not contain less than ten per cent. of saponifiable animal oil.

Special Mixture.

All special mixtures of oil not coming under the above specifications will be purchased on sample, which must be of one gallon. Shipments will be required to conform to sample in every particular. Samples must be sent as the purchasing agent may direct.

Chicago, Burlington and Quincy Railroad Co. Chemical Laboratory.

Aurora, Ill.,………………18..

To………………………………Supt. M. P.:

Dear Sir: I have made an examination of sample of above oil, and have obtained the following results:

Flashing point ……………°F. Ash…………%. Tar…….%.
Burning " ……………°F. Cold test…………at……°F.
Specific gravity……………°B. Viscosity at………°F. 100 cc. oil
Loss at …. °F. for 3 hours……%. flows from instrument in..seconds.

FRICTION TEST ON THE THURSTON OIL TESTER.

Date.	1st trial.		2d trial.		3d trial.		Average.
Amount used……………oz.	Temp.	Arc.	Temp.	Arc.	Temp.	Arc.	
Highest reading………………	……	……	……	……	……	……	……
Lowest " ………………	……	……	……	……	……	……	……
Range of " ………………	……	……	……	……	……	……	……
Average " ………………	……	……	……	……	……	……	……
Time run in minutes……………	…………		…………		…………		……
Total revolutions………………	…………		…………		…………		……
Revolutions per minute…………	…………		…………		…………		……
Speed, miles per hour…………	…………		…………		…………		……
Pressure, total lbs……………	…………		…………		…………		……
" lbs. per sq. inch………	…………		…………		…………		……
Coefficient of friction…………	…………		…………		…………		……
Lubricating value, with Extra Lard Oil as 100………………	…………		…………		…………		……

Received......................18..
Tested........................18..
Sample No. or Letter..............
Blank No..........................
Letter Book No........ Page......

Car No. and Initials................
Tank or No. Bbls...................
Name of firm supplying............
Price....cents per gallon.

Yours truly,

.............................Chemist.

For the..............................R. R. Co.

CHICAGO, BURLINGTON AND QUINCY RAILROAD COMPANY.

Specifications for Black Engine Oils.

("Petroleum lubricating oils;" "well oils;" "petroleum stock oils;" or "passenger and freight car lubricating oils.")

Uses.—For lubricating the journals of passenger and freight cars and locomotives, and for miscellaneous lubrication.

Grades.—: "Summer," "25 degree," "15 degree" and "zero."

Requirements.—For all grades:

Specific gravity, between 26° and 29° B.

Loss at 100° F. for three hours, not over one-fourth per cent.

Flashing point, for all but "zero" oil, not under 300° F.

Flashing point, for "zero" oil, not under 250° F.

Burning point, for all but "zero" oil, not under 375° F.

Burning point, for "zero" oil, not under 300° F.

Cold Test—Summer oil must flow at 60° F. or above.
" — 25° " " " 30° " "
" — 15° " " " 20° " "
" — Zero ." " " 5° " "

All these oils must be pure petroleum oils, free from other compounds, and from dirt, grit, lumps and specks; transparent and greenish or reddish (not black) in *tint*, when spread as a *thin* film on glass and looked through toward the light; translucent and greenish when held in a horizontal position. Preference will be given to those oils which are low in tarry matters and in ash, and which do not "froth" when tested for flash and fire.

Oils differing notably from above requirements will be rejected.

CHICAGO, BURLINGTON AND QUINCY RAILROAD COMPANY.

Specifications for Cylinder Stock.

Use.—For making cylinder lubricant. One grade.

Requirements.—Must have a flashing point not lower than 475° F., a burning point not lower than 575° F., and a specific gravity between 25° and 28° B. Must not undergo a loss greater than one-half (½) per cent., when exposed for three (3) hours to a temperature of 350° F. Must be free from dirt, grit, lumps and specks; transparent and greenish or reddish (not black) in tint, when spread as a thin film on glass and looked toward the light.

References: "Measurements of Friction of Lubricating Oils." By C. J. H. Woodbury, *Trans. Am. Soc. Mech. Eng.*, 6, 136.

"On the Theory of the Finance of Lubrication and on the Valuation of Lubricants by Consumers." By R. H. Thurston, *Trans. Am. Soc. Mech. Eng.*, 7, 437.

"Cost of Lubricating Car Journals." By L. A. Randolph, *Trans. Am. Soc. Mech. Eng.*, 10, 126-35.

"Special Experiments with Lubricants." By J. E. Denton, *Trans. Am. Soc. Mech. Eng.*, 12, 405-50.

"Report of Committee on Lubrication of Cars to the Master Car Builder's Association of the United States for 1893." *The Railway Car Journal*, 4, 156. (July, 1894.)

"History of Attempts to Determine the Relative Value of Lubricants by Mechanical Tests." *Proceedings of the American Association for the Advancement of Science*, 34.

"Car Lubrication." By W. E. Hall.

XLV.

Oils Used for Illumination.

Oils used for illumination may be classified into two groups:

1. Refined products from petroleum, such as naphtha, gasoline, kerosene, signal oil, etc.

2. Certain refined oils of vegetable and animal origin, as colza oil, rape oil, lard oil, sperm oil, etc.

1. Refined Products from Petroleum.

Kerosene is the refined product from petroleum that distills over (in the refining process) after the lighter oils, naphthas, etc., have been separated, and is the principle oil in use for illumination. In color it varies from standard white to water white (colorless), and its commercial value is dependent upon its flash and burning point. In the oil trade, the burning or fire tests are classified as 110° F., 120° F. and 150° F., and 300° F.

The 150° F. is known as headlight oil and the 300° F. as mineral sperm and mineral colza.

The requirements for mineral oils to be used in railroad illumination are as follows:

Specifications for Petroleum Burning Oils.

(Conditions of shipment and General Specifications.)

This material will be purchased by weight. Barrels must be in a good condition and must have the name of the contents and the consignee's name and address on each barrel, and plainly marked with the gross and net weight which will be subject to the Company's weight.

When received all shipments will be promptly weighed. If not practicable to empty all barrels, ten per cent. (10 %) will be emptied, and the losses of the whole shipment will be adjusted in accordance with the ten per cent. taken. Should the net weight thus obtained be less by one per cent. (1 %) than the amount charged in the bill, a reduction will be made for all over one per cent.

Prices should be given in cents or hundredths of a cent per pound.

Shipments, one or more barrels of which are filled with oil cloudy from the presence of glue, or which contain dirt, water or other impurities, will be rejected.

Two kinds of petroleum burning oils will be used, known as 150° fire test for general use, and 300° fire test for use in passenger cars.

Detail Specifications.

150° Fire Test Oil.

This oil must conform to the following requirements:

1—It must have a flash test above 125° F.
2—It must have a fire test not below 150° F.
3—It must have a cloud test not above 0° F.
4—It must be a "water white" in color.
5—Its gravity must be between 44° and 48° B. at 60° F.

300° Fire Test Oil.

This oil must conform to the following requirements:

1—It must have a flash test above 250° F.
2—It must have a fire test not below 300° F.
3—It must have a cloud test not above 32° F.
4—It must be a "standard white" in color.
5—Its gravity must be between 38° and 42° B. at 60° F.

Method of Making Tests.

150° Fire Test Oil.

The "Open Tagliabue" cup is used for determining the flashing and burning points of this oil, heating the oil at the rate of 2° F. per minute and applying the test flame every degree from 120° for flash and every 4° after flash for the burning point.

300° Fire Test Oil.

The "Cleveland" cup is used for determining the flashing and burning points of this oil, heating at the rate of 5° per minute and applying the test flame every 5° from 230° F.

Cloud Test.

The cloud test is made as follows: Two ounces of the oil are placed in a four ounce sample bottle, with a thermometer suspended in the oil. The bottle is exposed to a freezing mixture of ice and salt and the oil stirred with the thermometer while cooling. The temperature at which the cloud forms is taken as the cloud test.

The requirements for the flash and fire test for illuminating oils used for domestic purposes are not so rigid as for railroad practice. In fact large quantities of oil, flashing below 110° F. are used, the cheaper price being the incentive. So dangerous are these oils with low flash points, that many states have passed stringent laws against their use. An oil with a fire test of 110° F. very often has a flash test of 90° F. and many oils with a fire test of 120° F., flash at or below 100° F. It is the flash point of an oil that makes it dangerous and while the refiners of oils mark their products by the fire test, the laws as passed by many of the states, specify the flash test as the requisite.

There is no absolute ratio between the flash and fire test of an oil, since while many illuminating oils have a high fire and flash test, others may have a high fire and a low flash test.

The instrument that gives the best satisfaction in testing illuminating oils, not lubricating oils (see page 403), for the flash and fire test is called the Wisconsin Tester. (Fig. 143.)

It is thus described:

On the left side of the figure is shown the instrument entire. It consists of a sheet-copper stand eight and one-half inches high, exclusive of the base, and four and one-half inches in diameter. On one side is an aperture three and one-half inches high, for introducing a small spirit-lamp, *A*, about three inches in height, or better, a small gas burner in place of the lamp when a supply of gas is at hand. The water-bath, *D*, is also of copper, and is four and one-eighth inches in height and four inches inside diameter. The opening in the top is two and seven-eighths inches in diameter. It is also provided with a one-fourth inch flange which supports the bath in the cylindrical stand. The capacity of the bath is about twenty fluid ounces, this quantity being indicated by a mark on the inside. *C* represents the copper oil-holder. The lower section is three and three-eighths inches high, and two and three-fourths inches inside diameter. The upper part is one inch high and three and three-eighths inches in diameter, and serves as a vapor-chamber. The upper rim is provided with a small flange which serves to hold the glass cover in place. The oil holder contains about ten fluid ounces, when filled to within one-eighth of an inch of the flange which joins the oil cup and the vapor-chambers. In order to prevent reflection from the otherwise bright surface of the metal, the oil-cup is blackened on the inside by forming a sulphide of copper, by means of sulphide of ammonium.

The cover, *C*, is of glass, and is three and five-eighths inches in diameter; on one side is a circular opening, closed by a cork through which the thermometer, *B*, passes. In front of this is a second opening three-fourths of an inch deep and the same in width on the rim, through which the flashing jet is passed in testing. The substitution of a glass for a metal cover more readily enables the operator to note the exact point at which the flash occurs. A small gas jet, one-fourth inch in length, furnishes the best means for igniting the vapor. Where gas cannot be had the flame from a small waxed twine answers very well.

(2). The test shall be applied according to the following directions:

Remove the oil cup and fill the water-bath with cold water up to the mark on the inside. Replace the oil cup and pour in enough oil to fill it to within one-eighth of an inch of the flange joining the cup and the vapor-chamber above. Care must be taken that the oil does not flow over the flange. Remove all air bubbles with a piece of dry paper. Place the glass cover on the oil cup, and so adjust the thermometer that its bulb shall be just covered by the oil.

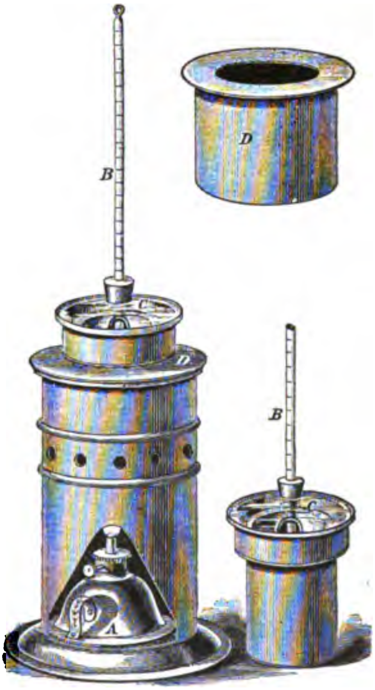

Fig. 143.

If an alcohol lamp is employed for heating the water-bath, the wick should be carefully trimmed and adjusted to a small flame. A small Bunsen burner may be used in place of the lamp. The rate of heating should be about 2° per minute, and in no case exceed 3°.

As a flash torch, a small gas jet, one-fourth inch in length, should be employed. When gas is not at hand, employ a piece of waxed linen twine. The flame in this case, however, should be small.

When the temperature of the oil has reached 85° F., the testings should commence. To this end insert the torch into the opening in the cover, passing it in at such an angle as to well clear the cover, and to a distance about half way between the oil and the cover. The motion should be steady and uniform, rapid and without any pause. This should be repeated at every 2° rise of the thermometer until the temperature has reached 95°, when the lamp should be removed and the testings should be made for each degree of temperature until 100° is reached. After this the lamp may be replaced, if necessary, and the testings continued for each 2°.

The appearance of a slight bluish flame shows that the flashing point has been reached.

In every case note the temperature of the oil before introducing the torch. The flame of the torch must not come in contact with the oil.

The water-bath should be filled with cold water for each separate test, and the oil from a previous test carefully wiped from the oil cup.

(3). The instrument to be used in testing oils which come under the provisions of section 2 of the law shall consist of the cylinder *D*, and the copper oil cup *C*, together with a copper collar for suspending the cup in the cylinder, and an adjustable support for holding the thermometer.

(4). The test for ascertaining the igniting point shall be conducted as follows: Fill the cup with the oil to be tested to within three-eighths of an inch of the flange joining the cup and the vapor-chamber above. Care must be taken that the oil does not flow over the flange. Place the cup in the cylinder and adjust the thermometer so that its bulb shall be just covered by the oil. Place the lamp or gas burner under the oil cup. The rate of heating should not exceed 10° a minute below 250° F., nor exceed 5° a minute above this point. The testing flame described in the directions for ascertaining the flashing point should be used. It should be applied to the surface of the oil at every 5° rise in the thermometer, till the oil ignites.

The following is a copy of the law of the state of New York regulating the standard of illuminating oils, etc.:

AN ACT to regulate the standard of illuminating oils and fluids for the better protection of life, health and property.

Passed June 6, 1882, three-fifths being present.

SECTION 1. No person, company or corporation shall manufacture or have in this State, or deal in, keep, sell or give away, for illuminating or heating purposes in lamps or stoves within this state, oil or burning fluid, whether the same be composed wholly or in part of naphtha, coal oil, petroleum or products manufactured therefrom, or of other substances or materials, which shall emit an inflammable vapor which will flash at a temperature below one hundred degrees, by the Fahrenheit thermometer, according to the instrument and methods approved by the State Board of Health of New York.

§ 2. No oil or burning fluid, whether composed wholly or in part of coal oil and petroleum or their products, or other substance or material, which will ignite at a temperature below three hundred degrees by the Fahrenheit thermometer, shall be burned in any lamp, vessel or other stationary fixture of any kind, or carried as freight, in any passenger car, or passenger boat moved by steam power in this State, or in any stage or street car drawn by horses. Exceptions as regards the transportation of coal oil, petroleum and its products are hereby made when the same is securely packed in barrels or metallic packages, and permission is hereby granted for its carriage in passenger boats moved by steam power when there are no other public means of transportation. Any violation of this act shall be deemed a misdemeanor and subject the offending party or parties to a penalty not exceeding three hundred dollars, or imprisonment not exceeding six months, at the direction of the court.

§ 3. It shall be the duty of the State Board of Health of New York to recommend and direct the nature of the test and instruments by which the illuminating oils, as hereinbefore described, shall be tested in accordance with this act. It shall be the duty of the public analysts, who may now be employed by the State Board of Health, or who may be hereafter appointed, to test such samples of suspected illuminating oils or fluids as may be submitted to them under the rules to be adopted by the said board, for which service the said board shall provide reasonable compensation at the first quarterly meeting of the State Board of Health after the passage of this act; it shall adopt such measures as may seem necessary to facilitate the enforcement of this act, and prepare rules and regulations with regard to the proper methods of collecting and examining suspected samples of illuminating oils, and the State Board of Health shall be authorized to expend, in addition to all sums already appropriated for said board, an amount not exceeding five thousand dollars for the purpose of carrying out the provisions of this act. And the sum of five thousand dollars is hereby appropriated, out of any moneys in the treasury not otherwise appropriated, for the purposes of this section as provided.

§ 4. Naphtha and other light products of petroleum which will not stand the flash test required by this act may be used for illuminating or heating purposes only.

In street lamps and open air receptacles apart from any building, factory or inhabitated house in which the vapor is burned.

In dwellings, factories or other places of business when vaporized in secure tanks or metallic generators made for that purpose in which the vapor so generated is used for light or heating.

For use in the manufacture of illuminating gas in gas manufactories, situated apart from dwellings and other buildings.

§ 5. It shall be the duty of all district-attorneys of the counties in this State to represent and prosecute in behalf of the people, within their respective counties, all cases of offenses arising under the provisions of this act.

§ 6. Nothing in this act shall be so construed as to interfere with the provisions of chapter seven hundred and forty-two of the laws of eighteen hundred and seventy-one, as regards the duties of the Bureau of Combustibles of the city of New York, or any other statutes not conflicting with this act, provided that nothing in this act shall be deemed to interfere with or supersede any regulation for the inspection and control of combustible materials in any city of this State made and established in pursuance of special or local laws or the charter of said city.

§ 7. All acts or parts of acts inconsistent with this act are hereby repealed.

§ 8. This act shall take effect sixty days after its passage.

A very complete report upon the methods and apparatus for testing inflammable oils by A. H. Elliott, Ph.D., was rendered to the New York State Board of Health and incorporated in their annual report for 1882.

The grades of color of an oil are noted as standard white, prime white, superfine white and water white,[1] and the instrument generally used for determination of the color in oils, is the Stammer Colorimeter (Fig. 144). Tube *I* is closed at the bottom by a transparent glass plate, is open at the top, and a projecting lip on the side whereby the oil to be tested can be poured in or out. The tube is fastened to the stand by two screws. The measuring tube *III* is closed at the bottom by a colorless glass plate and is movable inside of tube *I*.

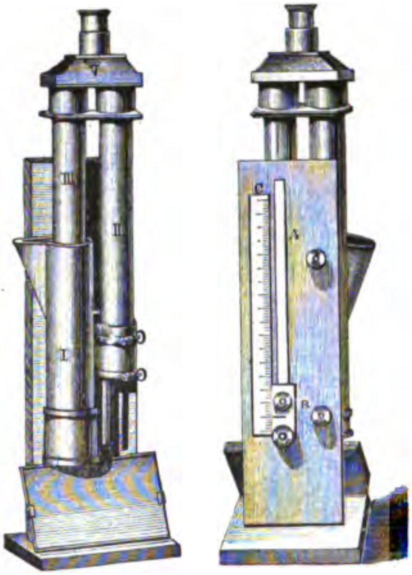

Fig. 144.

The color-glass tube *II* which is joined firmly to the measuring tube *III*, is open at the bottom and at the top contains a colored glass plate, which plate can be substituted with other tinted glass plates. The movement of the joined tubes *II* and *III* is produced by inclosed racket work, the movement of the

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[1] In Bremen, the varieties are rated as, water white, prime white, standard white, prime light straw, light straw and straw.

tubes being read on a scale on the back of the stand, and stated in millimeters. Since the color of a liquid is inversely proportional to the height of the column, which is necessary to give the standard color, and since this color is here expressed by 100, the absolute number for expressing the tone of color of any oil is obtained by dividing this 100 by the number of millimeters read off from the scale. For example:

Millimeter scale = 1. Color = 100.00
" " = 2. " = 50.00
" " = 7. " = 14.29
" " = 19. " = 5.26

The color, tone and thickness of the standard glass is so chosen that the scale shows the following values for the ordinary brands of illuminating oils.

Standard white = 50.0 mm.
Prime " = 86.5 "
Superfine " = 199.5 "
Water " = 300.00 "

Wilson's calorimeter, largely used in England, is very similar in construction to the Sterner.

2. *Vegetable and Animal Oils.*

The two principal oils of this class in use for illumination are colza and lard oil.

In this country the former has never been used to any great extent, its use being confined principally to Europe, but lard oil and sperm oil, in former years, before the introduction of the petroleum products for this purpose, were largely used as illuminants. Except in railroad practice and then in yearly decreasing amounts their use now is very limited in this direction. In the matter of illumination, the methods made use of by the railroads are worthy of study and comparison, and it is in a great measure due to the investigations carried out in their interests that the great advances in this direction are due.

George Gibbs, mechanical engineer of the Chicago Milwaukee and St. Paul Railroad, in a paper recently read before the Western Railroad Club of Chicago, states:

There are about eight different means of car illumination; *viz.*, the use of

1. Vegetable oils. 2. Candles. 3. Mineral or petroleum oils. 4. Ordinary coal gas. 5. Carburetted water gas. 6. Rich or oil gas. 8. Electric light. There are but four worthy of consideration. These are:

First. *Heavy mineral oils in lamps*, such as mineral seal which ranges from 35° B. to 40° B. in gravity, has a fire test of 300° F. and gives off no inflammable vapor below 230° F.

Second. *The Pintsch oil gas*. This is by far the most prominent attempt to devise any economical and practical gas-lighting system for railroad service. Its primary object is to reduce the bulk of stored gas necessary to produce an adequate illumination for a considerable length of time.

The Pintsch system has largely confined its attention to more efficient gas, which, it is claimed, is supplied by the use of a rich permanent oil gas.

Ordinary city or coal gas when burned at pressure of the street mains, one to one and one-half ounces may be taken to give an illumination of, at most, four candles per cubic foot. Oil gas at the same pressure will give from four to six times as much, say sixteen candles per cubic foot. But one property of gas, which vitally affects the problem, is the loss of light-giving power upon compression and storage. This is true of all illuminating gas, and is due to the deposition of the rich oily hydrocarbons, but is not true to the same extent for oil and coal gas, the difference being materially in favor of oil gas. Reliable tests for this loss of light by compression have given the result that coal gas loses fifty per cent. and oil gas twenty-one per cent. of light-giving power upon compression of 300 pounds per square inch, and at 225 pounds per square inch pressure, the quantities required for equal illumination would be about as five of coal to one of oil gas.

The material used for the manufacture of Pintsch gas is crude petroleum. The generation of gas is affected by vaporizing the oil at a high heat in suitably arranged cast iron retorts, the process of manufacturing being, on a small scale, essentially that followed for city gas. From the storage tank pipe connections lead to convenient places for filling the car tanks. A plant capable of making sufficent gas for 500 cars is contained in a one story building 26 ft. × 36 ft. The outfit on the cars consists of one or two cylinders for holding the compressed gas, a pressure regulator and a system of piping to the lamps. These are of special design, each having from four to six flames arranged beneath a procelain reflector, the whole encased in a glass bell-jar; ventilation is suitably provided for and a very steady light is obtained.

Mention might be made here that an American system, the Foster, appeared a few years ago embodying the same principles and general features as the Pintsch.

Third. *The Frost Systems.* In the Frost and all other similar systems the principle is the same, being the property possessed by air of holding a vapor in intimate mixture and suspension, usually the vapor of gasoline. The amount of vapor absorbed depends upon its temperature; thus, at 14° above zero (F.), about six per cent. and at 68° F., twenty-seven per cent. will be taken up. This is, however, a mechanical mixture only and not a permanent gas. The vapor thus formed is capable of being burned similarly with gas, when mixed with air in the proper proportions, giving a highly luminous flame. This principle has been utilized for many years for making gas for household purposes in places where city gas is inaccessible, a simple form of air pump run by a falling weight forcing air under a few ounces pressure through a tank (generally under ground) which contains a barrel of liquid gasoline. This tank is divided into many compartments in which absorbent wicking is suspended, dipping into the liquid and drawing up the same by capillary attraction. The enriched air produced in this carburetter forms the gas for burning.

The difficulties to be overcome in using this agent for safe car lighting are as follows: First, the presence of liquid gasoline. The Frost system overcomes this objection by filling the carburetting vessel almost completely with wicking and by merely saturating this with gasoline and drawing off the superfluous liquid. Second, the effect of variation of temperature in the amount of vapor absorbed by the air current. As above stated, in cold weather only a small percentage is absorbed, too little to produce a good light and in warm weather too much, producing a rich but smoky light. This is really the serious stumbling block to this system. The Frost system claims to overcome it by placing a small generator or carburetter above the light on the roof of the car, in such a manner that a portion of the heat generated by the burner is transmitted to the carburetter, insuring a uniform temperature at all times.

The system in detail consists of an air storage tank underneath the car, containing sufficient compressed air to supply light for six hours. This compressed air is obtained directly from the train pipe of the air brake and is led through a suitable pressure reducer and a regulator to the carburetters in the roof, one of these being placed over each lamp, and thence, after passing through them, to the lamps underneath. These are now constructed on the Siemans or regenerative principle and give a brilliant white light without shadow. The supply of gasoline in the carburetters is sufficient for forty-three hours burning, and then can be recharged by filling from the roof.

Fourth. *The Electric System*. The latest phase of train lighting may be said to be the electric. In this direction numerous isolated experiments have been made in this country during the past five years. The different plans suggested for obtaining electric lighting are divided as follows:

1. Primary batteries;
2. Secondary batteries or accumulators;
3. Dynamo machine connected to car axle, with or without accumulators as auxiliaries;
4. Dynamo operated by special steam engine, either in a car or on the locomotive, and supplied with steam from locomotive or special boiler on a car: accumulators used or not, as desired, as equalizers.

1. The first method has been tried in England on several railways, and in France between Paris and Brussels. In all, a special form of primary battery having very low resistance, great surface, and furnishing a constant current at high pressure, was employed. The result was flat failures, on account of the enormous expense of the electrical energy furnished by chemical means. It can be said that in primary batteries chemicals are expended and zinc consumed, instead of coal under a boiler to

produce energy; at the lowest estimate, the former is forty times as expensive as the latter.

2. In England the London and Brighton railway made an extensive trial on a Pullman train of lighting by accumulators alone, placing batteries under each car, and having a sufficient number of charging stations, with boilers, engines and dynamos, to charge duplicate sets of batteries for immediate replacement.

This system, after five years trial, was abandoned. In this country the Pullman Company gave the method a thorough trial on the Pennsylvania Railroad "Limited" between New York and Chicago, finally abandoning it. It was also tried and abandoned on the Baltimore and Ohio and Chicago, Burlington and Quincy. Description of this system may be dismissed by briefly stating that each car carries its own store of batteries in boxes hung underneath, arranged so that they can be readily removed at terminals for recharging by dynamo, or for substitution of fresh cells. The weight of batteries required for a standard coach is, approximately, one ton.

3. Third method. A favorite method for obtaining electricity at a low cost seems to have been to connect the dynamo to a car axle; but the difficulties of obtaining regular motion and current and providing light when the train stops, have necessitated the employment of accumulators as regulators and auxiliaries. In these, automatic appliances are provided to cut off the current from the dynamo when the speed of the train falls below a certain rate, and to deliver the current to the batteries in the same direction. The main difficulty, with this method, and one which the International Railway Congress states has not been solved satisfactorily, is the transmission of power from the axle to the dynamo.

4. The fourth method is the only prominent electrical one in this country for car lighting. It consists essentially in the use of a dynamo driven by special steam engine, with secondary batteries for reserve. The use of the method without the batteries as auxilaries has been often attempted without success, but recently by improvements made by Mr. Gibbs, the batteries are dispensed with and a system perfected that gives general satisfaction for the purpose. The plant in fact is made an exact

duplicate of stationary electric-lighting plants. The engine is a 15 horse-power Westinghouse automatic, the dynamo a 150 light Edison compound-wound, connection from one to the other being made by belting. In the summer season, when steam heat is not required for the train, this outfit is placed in the forward end of the baggage car, occupying twelve feet in the length of the car, but not obstructing passageway through it. Steam is taken, at sixty pounds pressure, from the locomotive boiler. In winter the drain upon the locomotive for steam heat is often excessive; to overcome this a special car for heating and lighting is used. Consult *Engineering* (London), January 5, 1894, for a complete description of this system as now used successfully on the Chicago, Milwaukee and St. Paul Railroad.

Relative Advantages and Disadvantages of the Various Systems.

The Electric may be considered adapted, in the present state of the art, to special service only. It fills a number of the requirements for a perfect light in a manner that no other light approaches; it is cleanly, cool, safe, allows excellent distribution and is, in fact, a luxury which is duly appreciated by the public. It, however, is costly, and requires great attention to details; still, in many instances it will pay, and each manager must consider whether under his conditions its use is warranted.

The Frost System is still in the process of development. It has many advantages from an outside point of view; it is cleanly, the light is good, each car is perfectly independent of others for its supply of light, and it requires no external gas works. On the other hand the first cost is excessive; the light is not cheap for running, its quality is not uniform—due to the effect of varying temperature and quality of gasoline—the apparatus is complicated, and while the system may be considered safe to the car itself, the use of gasoline at various points on a large system is questionable.

The Pintsch System. This, in spite of some defects, is probably the most feasible and promising method in the direction of safety car lighting. It is safe as any flame method of lighting can be, is cleanly and simple, and is cheap in maintenance and running.

It is, however, very high in first cost, and is not universally applicable on account of dependence upon gas works. But all main line traffic and many important branch lines can generally be provided for by this system at a moderate cost and under its rapid extension now taking place, it seems likely that gas works can be maintained by different roads at many points, to still further reduce the individual outlay.

Oil Lighting by Lamps. Many of the requirements of a satisfactory car-lighting system appear to be embodied in the present oil system, or might be with some improvements which are readily attainable. In no system, with the exception of the electric, is it possible to obtain a better or more satisfactory distribution of light, the centers being of moderate intensity; the fuel is safe to handle and may be obtained without delay; each car is independent of the others; it is cheapest in first cost and maintenance for a given amount of light; it is simple and requires but little attention. On the other hand, it shares with flame systems the objections of giving out much heat; the quantity of light is often irregular and the smell objectionable when proper care is not exercised.

The possible improvements in this system should have more attention from railway officials.

For instance, the button form of burners, of which the "Acme" is a good example, appears to solve the problem of sufficient light as has been done in the other flame systems, and these burners should be substituted for the old uneconomical forms.

COMPARATIVE COST OF CAR LIGHTING SYSTEMS.

Figures based on service for one 50 foot passenger coach.

System.	No. of burners.		Candle power of light.			First cost of equipment.	Depreciation and five per cent. interest on cost.	Cost per car per H. P.			Cost per candle power per hour. Cents.	Hours burning without refilling.	Cost of equipment per candle power.	Price of oil or fuel.
	Main body of car.	Closet.	Per burner in body of car.	Total in body of car.	Entire car.			Oil. Cents.	Supplies and attendants. Cents.	Total running. Cents.				
No. 1. Colza oil	12	1	12	144	156	$167.00	$16.70	12.22	1.06	13.28	0.085	14	$1.07	65 cts. per gallon.
No. 2. Mineral oil, (Mochring burner)	12	1	12	144	156	167.00	16.70	1.67	1.06	2.73	0.018	15	1.07	9½ cts. per gallon.
No. 3. Mineral oil, (Duplex burners.)	12	1	10.5	126	136.5	167.00	16.70	1.21	1.06	2.27	0.017	18	1.22	9½ cts. per gallon.
No. 4. Mineral oil, "Acme" burners	6	1	26	156	168	113.00	11.30	1.94	.57	2.51	0.015	13	0.67	9½ cts. per gallon.
No. 5. Pintsch gas	16	1	10	160	170	552.00	64.07	2.05	.55	2.60	0.015	34	3 24	$1.87 per 1000 cubic ft.
No. 6. " Frost " carburetted air	4	1	42	168	180	536.00	80.40	3.48	1.12	4.60	0.026	43	2.98	15 cts. per gallon.
No. 7. Elect'c storage and direct current	9	1	16	144	160	863.00	137.47	....	....	13.20	0 083	trip.	5.40	
No. 8. "Gibbs" electric, direct	9	1	16	144	160	395.40	36.35	....	.. .	9.83	0.061	"	2.47	

XLVI.

The Analysis of Lubricating Oils Containing Blown Rape-Seed and Blown Cotton-Seed Oils.

Rape-seed oil has long been the standard oil in Europe for lubrication. Its constancy of viscosity at varying temperatures, its non-liability to acidity as compared with other seed oils, and its low cold test, unite in producing the results required of a good lubricant. It, however, is no exception to the rule that vegetable and animal oils suffer partial decomposition when subjected to high temperature produced by friction, with the result that fatty acids are liberated and corrosion of bearings produced.

The substitution of mineral oils in varying proportions with rape-seed oil has reduced this tendency, this reduction being determined by the percentages of mineral oil present, as the latter liberates no free acids.

It is a peculiar fact, however, that a mineral oil alone does not give as satisfactory results in lubrication (especially cylinder lubrication[1]) as does a mixture of mineral and vegetable or mineral and animal oils, one of the primary causes being that the viscosity of mineral oils rapidly diminishes at high temperatures, whereas the reduction of viscosity of vegetable and animal oils is very much less.

If it were not for this peculiarity between these two classes of oils, mineral lubricating oils could easily supplant (on the score of cheapness) all other oils used in lubrication.

The admixture of oils then being required for the better class of lubricants, it follows that in England where rape-seed oil has been the standard, its use should be continued in compounded oils.

The proportion of rape-seed oil added to mineral oil varies from five to twenty per cent. Where the mineral oil is a clear paraffin oil twenty per cent. of the seed oil is used; where the mineral oil is a dark, heavy oil, five per cent. is generally added.

The separation and estimation of the rape-seed oil in these mixtures presents no difficulty to the analytical chemist when no other seed oil is present, since the saponification of the seed oil,

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[1] *The Railroad and Engineering Journal*, 64, 73-126.

the separation of the fatty acids and recognition of the same are a part of the usual chemical work of this character. The recognition of the constituents of a mixed lubricating oil by analysis is a very different problem from giving a formula by which the mixture can be made. This is evidenced as follows:

Suppose the analysis shows

Rape-seed oil, 20 per cent.
Paraffin oil, 80 per cent.

Paraffin oil varies in specific gravity from 0.875 to 0.921, and it is essential to include in the report of the analysis not only the amount of the paraffin oil but also the gravity, since paraffin oil of gravity 0.875 is a very different product from that of 0.921 gravity, the former selling at seven and one-half cents and the latter at twenty-three cents per gallon. This determination can be made by taking the gravity of the original mixed oil (0.912), then knowing by the analysis that twenty per cent. is rape-seed oil (gravity 0.918), the gravity of the eighty per cent. of paraffin oil is easily calculated. Thus:

$x =$ specific gravity of rape-seed oil (0.918)
$y =$ specific gravity of paraffin oil
$x =$ 20 per per cent. or $\frac{1}{5}$
$y =$ 80 per cent. or $\frac{4}{5}$
Then $\frac{1}{5}x + \frac{4}{5}y = 0.912$
$0.183 + \frac{4}{5}y = 0.912$
$\frac{4}{5}y = 0.729$
$y = 0.910$

The mixture being composed, therefore, of
Paraffin oil (sp. gr. 0.910), 80 per cent.
Rape-seed oil (sp. gr. 0.918) 20 per cent.

The direct determination by analysis from the ether solution of the mineral oil in the mixture does not give an oil of the same specific gravity as the mineral had before it was mixed with the seed oil. This can be accounted for by the volatilization of a portion of the lighter hydrocarbons of the mineral oil when the ether is expelled during the analysis. For this reason the determination of the percentage of seed oil and the calculation of the mineral oil offers less liability to failure than finding the mineral oil directly.

The introduction of blown rape-seed oil instead of the normal rape-seed oil complicates the investigation and renders the use of the formula above given, valueless. Rape-seed oil has a gravity of 0.915 to 0.920. Rape-seed oil blown has a gravity of from 0.930 to 0.960.

Two difficulties are immediately presented: (1) The chemical analysis does not indicate whether the rape-seed oil is blown or not; (2) The use of the formula given without the correct gravity of the blown oil would give false results regarding the paraffin oil. To overcome this difficulty some synthetical work is required.

Suppose the specific gravity of the mixed oil is 0.922 and the analysis shows twenty per cent. of rape-seed oil. It will be necessary then to produce a mixture in these proportions that will duplicate the original sample. A check upon this will be the viscosity of the original sample as compared with the one to be made by formula. Thus:

The original oil has a gravity of 0.922, contains (by analysis) twenty per cent. of rape-seed oil, and has a viscosity at 100° F. of 335 seconds (Pennsylvania Railroad pipette).

First.—Make a mixture of paraffin oil (sp. gr. 0.910) generally used in this character of lubricant, eighty per cent., and rape-seed oil (unblown) twenty per cent. The viscosity is 165 seconds, showing that this mixture cannot be used in place of the original oil.

Second.—Make a mixture of paraffin oil (sp. gr. 0.910) and rape-seed oil partially blown, (sp. gr. 0.930) in the same proportions as above. The resulting viscosity is 267 seconds, showing that the compound is still lacking in viscosity.

Third.—Make a mixture of paraffin oil (sp. gr. 0.910) eighty parts, and rape-seed oil, blown (sp. gr. 0.960), twenty parts. The viscosity is 332 seconds.

This now fulfills the conditions required and the synthetical sample agrees with the original in gravity, composition and viscosity.

The use of blown rape-seed oil is being gradually replaced by blown cotton-seed oil. The latter, which has had but a limited use in lubrication, owing to its liability to acidity, has been greatly improved by this process of "blowing," which is nearly complete oxidation of the oil under comparatively high temperature.

This largely prevents the occurrence of the acidity in the oil, and thus the main objection to its use in lubrication disappears. It is much cheaper than rape-seed oil, since it costs thirty cents per gallon, to sixty cents per gallon for the latter. The chemical reactions of the two oils are very similar, and careful analytical work is required that the chemist be not misled.

The following table of comparisons will indicate this:

SPECIFIC GRAVITY.

Cotton-seed oil	0.920 to 0.925
Rape-seed oil	0.915 to 0.920
Blown cotton-seed oil	0.930 to 0.960
Blown rape-seed oil	0.930 to 0.960

VISCOSITY (PENNSYLVANIA RAILROAD PIPETTE) AT 100° F.

	Seconds.
Cotton-seed oil (sp. gr. 0.925)	162
Rape-seed oil (sp. gr. 0.918)	210
Blown cotton-seed oil (sp. gr. 0.960)	2143
Blown rape-seed oil (sp. gr. 0.960)	2160

HEIDENREICH'S TEST.

	Before stirring.	After stirring.
Cotton-seed oil	Faint reddish brown	Brown.
Rape-seed oil	Yellow brown	Brown.

MASSIE'S TEST.

Cotton-seed oil	Orange red.
Rape-seed oil	Orange.

IODINE ABSORPTION.

Cotton-seed oil	104 to 114
Blown cotton-seed oil	93 to 103
Rape-seed oil	102 to 108
Blown rape-seed oil	94 to 100

In the comparison of the two oils, when not mixed with a mineral oil, the above tests can be used. The conditions are altered, however, when either one or both are so mixed, since these tests apply only to the pure oils and not to those reduced with large percentages of mineral oil. After the separation of the seed oil from the mineral oil by saponification the identification of the seed oil depends upon the reactions of the fatty acids obtained, and a careful examination and comparison of these reactions shows that the melting points have the greatest difference and thus become a means of recognition.

Thus, the fatty acids from rape-seed oil melt at 20° C., and from cotton-seed oil at 30° C. Hence, if upon analysis of a lubricating oil under above conditions, the fatty acids obtained show a melting point of 20° C. the seed oil can be pronounced rape-seed oil.

If the melting point is between these limits, say 23° C., the seed oils are present in a mixture, the proportions of which can be determined by the following formula:

w_1 = proportion of rape-seed oil.
w_2 = proportion of cotton-seed oil.
w_3 = weight of mixture (20 per cent.)
t_1 = temperature of melting point of fatty acids of rape-seed oil.
t_2 = temperature of melting point of fatty acids of cotton-seed oil.
t_3 = temperature of melting point of mixed fatty acids.

Then $w_1 = w_3 \frac{t_3 - t_2}{t_1 - t_2}$

$$w_2 = w_3 \frac{t_3 - t_1}{t_2 - t_1}$$

Inserting the value:

$$w_1 = 20 \frac{23 - 30}{20 - 30} = 14 \text{ per cent.}$$

$$w_2 = 20 \frac{23 - 20}{30 - 20} = 6 \text{ per cent.}$$

Or,

Paraffin oil	80 per cent.
Rape-seed oil	14 per cent.
Cotton-seed oil	6 per cent.
Total	100 per cent.

By synthetical work upon these proportions, with comparison of viscosities of the sample submitted with the product, the result will be not only a correct analysis, but a working formula can be given by which a manufacturer can duplicate the original oil.

XLVII.

The Analysis of Cylinder Deposits.

The deposits in steam cylinders, formed by the decomposition of lubricating oils, may be classed as simple or compound, depending upon whether the deposit is due to the decomposition of the oil alone or if foreign matters, carried over in the steam from the boilers, are also present.

In the former case, carbon, hydrocarbons, oils and iron oxide are the principal constituents, whereas, in the latter, oleate of lime, carbonate of lime, and silica are often present in addition to the former.

The following analysis of a sample from a locomotive cylinder would indicate a simple deposit.

Moisture		2.28	per cent.
Oils soluble in ether	Animal	10.54	"
	Mineral	11.23	"
Hydrocarbons insoluble in ether		47.97	"
Fixed carbon		23.73	"
FeO		2.83	"
Undetermined		1.42	"
Total		100.00	"

And the one given below, of a deposit from the steam cylinders of a large stationary engine, would show that scale-forming material from the boilers had become a component.

Moisture		13.12	per cent.
Oils soluble in ether	Animal	8.15	"
	Mineral	7.86	"
Soap		2.10	"
Hydrocarbons insoluble in ether		1.67	"
Fixed carbon		2.71	"
Oxides of Iron and aluminum		6.81	"
SiO_2		3.65	"
$CaCO_3$		43.22	"
$MgCO_3$		10.17	"
Undetermined		0.44	"
Total		100.00	"

In many samples I have found copper and zinc in the deposits, formed by the corrosive action of the liberated oleic acid from the animal oil upon the brass or composition bearings.

This corrosive action is very marked where a poor quality of lubricating oil, composed of animal or vegetable oil, is used, whereas, a pure neutral mineral oil has no acid action at steam temperature. Oftentimes the statement has been made to me, when the deposit was given for analysis, "All of our lubricating oil is pure mineral oil; we use no other." And yet, upon analysis, lard oil would be shown in comparatively large amounts.

This is accounted for from the fact that while the consumer believes he is using pure mineral oil—which was sold to him as such—the manufacturer has introduced from three to thirty per cent. of lard or cotton-seed oil.

A large majority of the so-called "pure mineral" lubricating oils for cylinder use contain at least five per cent. of animal oil; and it is the exception and not the rule to find a "pure mineral" oil for cylinder lubricating purposes.

An analysis of a deposit from the steam cylinder of a large freight steamer gave as a result:

Moisture		16.16	per cent.
Oils soluble in ether	Castor oil	26.19	"
	Mineral	32.50	"
Fixed carbon		7.92	"
CuO		0.50	"
FeO		25.10	"
Undetermined		1.63	"
Total		100.00	"

Pure mineral lubricating oil was supposed by the officers of the vessel to be the only lubricant used, and special care had been taken to secure it, but it appears that the engineer added a small amount of castor oil to the mineral oil, as, in his opinion, it made a better lubricant.

The decomposition of the castor oil and liberation of the fatty acids was the primary cause of the deposit.

The action of the fatty acids upon the iron and metal bearings results in different products. That is to say, while the copper when present has generally been estimated as copper oxide the iron may exist only as oxide or as metallic iron, or both.

No doubt the oleic acid acts to form salts of these metals, but it is certain, in many instances, that when formed, they are immediately decomposed or partially so, and a resulting mixture formed that is somewhat difficult of analysis.

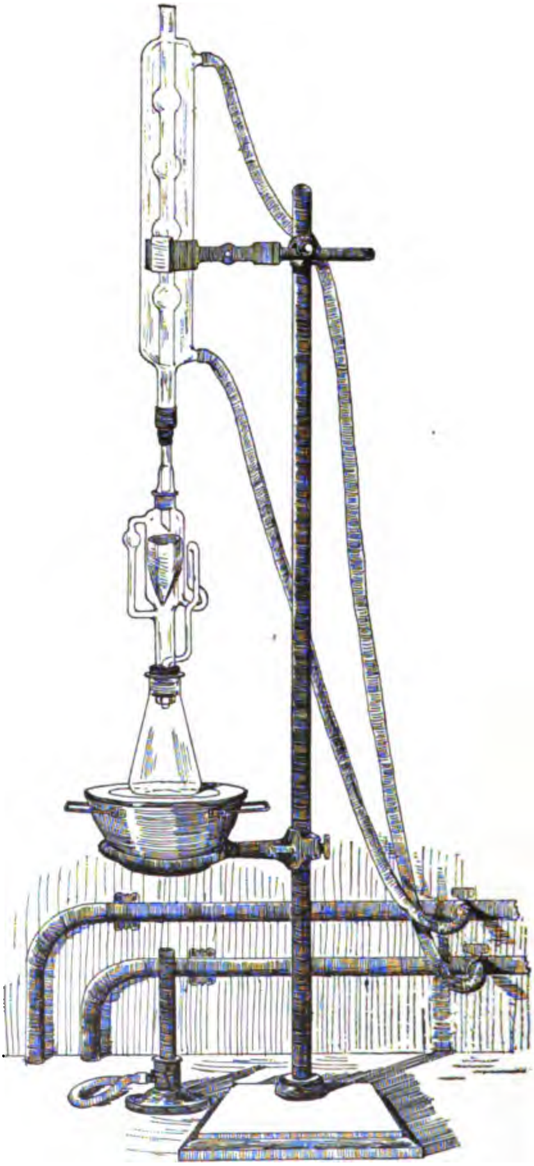

Fig. 145.

In the analysis here given, it will be noticed that the iron was found both as metal and as oxide.

Moisture		3.77	per cent.
Oils soluble in ether	Animal	21.27	"
	Mineral	19.60	"
Soap		traces	
Fixed carbon		10.90	"
FeO		14.01	"
Fe		27.85	"
PbO		0.82	"
CuO		1.07	"
Undetermined		0.71	"
Total		100.00	"

The evolution of hydrogen by hydrochloric acid, from the deposit, after all the oils and fatty substances had been removed, indicated the presence of metallic iron, and the analysis of the residue, after the combustion of the fixed carbon, gave figures by which the ratio of iron and iron oxide could be determined. A portion of the deposit, after extraction of oils by ether,[1] is dried, then weighed, the hydrocarbons driven off by heat, and the amount of fixed carbon present converted by combustion with sulphuric acid and chromium trioxide into carbon dioxide and weighed, this weight being calculated back to carbon.

Another portion of the same residue is ignited in a platinum crucible until the carbon is all consumed, then weighed. If the amount of carbon found is small and iron large, this weight may be larger than the original weight of the residue taken, owing to oxidation of metallic iron to ferric oxide.

Knowing the weight of carbon, and by making a determination of iron in another sample before ignition, the amount of iron oxide is easily found.

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[1] The Soxhlet apparatus as shown in Fig. 145 is well adapted for this purpose.

Scheme for Analysis of Cylinder Deposits.

<table>
<tr><td colspan="9">(1) Weigh out ten grams of the deposit upon a tared filter; dry at 102° C. to constant weight. Loss is moisture. The filter and contents are transferred to a Soxhlet tube, Fig. 145, and exhausted with ether.</td></tr>
<tr><td colspan="7">(2) Residue.—If qualitative analysis has shown soap to be present, the residue is dried slowly and then water poured upon the filter until extraction is complete.</td><td colspan="2" rowspan="3">(3) Solution.—Contains the free oils. Determine their weight by evaporation of the ether; then treat the residue with alcoholic solution of potash. Drive off the alcohol, treat the mass with warm water, transfer to a separatory funnel, allow to cool, add sufficient ether, agitate and set aside six hours.
Draw off the soap solution.</td></tr>
<tr><td colspan="6">(4) Residue.—Dry; take a weighed portion; transfer to a weighed covered platinum crucible, and ignite gently; air excluded. The loss represents volatile hydrocarbons insoluble in ether. Determine this, then ignite at lowest possible heat until weight is constant. (Accurate determination of fixed carbon requires oxidation with $H_2SO_4 + CrO_3$ to CO_2 and weighing as such, first treating with dilute HCl to drive off any carbonic acid united with CaO or MgO). Loss is fixed carbon. Residue contains the oxides of the metals, etc. The residue is weighed, then transferred to a porcelain capsule, HCl added with a few drops of HNO_3, and evaporated to dryness. Take up with HCl and water, filter; wash well.</td><td rowspan="6">(5) Solution.—Evaporate to dryness and determine weight of soap. Separate the fatty acids and test qualitatively for animal and vegetable oils.
CO_2 is determined by taking a weighed sample of residue marked (4) treating with HCl and weighing the CO_2 evolved.</td></tr>
<tr><td rowspan="5">(6) Residue.—Insoluble mineral matter. SiO_2, etc.</td><td colspan="5">(7) Solution.—Pass H_2S; filter.</td></tr>
<tr><td rowspan="4">(8) Residue.—Sulphides of copper and lead. Separate and determine their amounts and calculate to oxides of copper and lead.</td><td colspan="4">(9) Solution.—Boil out H_2S, oxidize with HNO_3, add ammonia to alkaline reaction; filter.</td><td rowspan="4">Soap Solution.—Warm gently for one-half hour, acidify with dilute H_2SO_4, allow separated fatty acids to collect and weigh with wax. Consult <i>J. Anal. Appl. Chem.</i>, 5, 710.</td><td rowspan="4">Ether Solution—Evaporate to dryness and determine weight of mineral oil. Test also for soaps insoluble in water, but soluble in ether. (Lead soap, zinc soap, lime soap, etc.)</td></tr>
<tr><td rowspan="3">(10) Residue.-$Fe_2O_3(Al_2O_3)$ Dry, ignite, and weigh as such, and calculate to FeO. If qualitative analysis has shown iron, as metal, determine the Fe also.</td><td colspan="3">(11) Solution.—Add H_2S to solution and filter.</td></tr>
<tr><td rowspan="2">(12) Residue. ZnS.—Dissolve in HCl and determine zinc as carbonate and calculate to zinc oxide.</td><td colspan="2">(13) Solution.—Boil out H_2S, add ammonium oxalate and a few drops of ammonium hydroxide: set aside six hours; filter.</td></tr>
<tr><td>(14) Residue, CaO.—Ignite and weigh as CaO.</td><td>(15) Solution.—Add solution sodium phosphate; set aside two hours; filter, dry, ignite and weigh as $Mg_2P_2O_7$. Calculate to MgO.</td></tr>
<tr><td>SiO_2.</td><td>CuO. PbO.</td><td>Fe, FeO, and (Al_2O_3).</td><td>ZnO.</td><td>CaO.</td><td>MgO.</td><td>CO_2.</td><td>Animal oil.</td><td>Mineral oil.</td></tr>
</table>

Where qualitative analysis has shown the deposit to be a simple one, the analysis can be stated as follows :

Moisture		———	per cent.
Oils soluble in ether	Animal	———	"
	Mineral	———	"
Hydrocarbons insoluble in ether		———	"
Fixed carbon		———	"
FeO		———	"
Fe		———	"
Total		———	"

For a complex deposit, the following form can be used :

Moisture		———	per cent.
Oils soluble in ether	Animal	———	"
	Mineral	———	"
Soap		———	"
Hydrocarbons insoluble in ether		———	"
Fixed carbon		———	"
Fe		———	"
FeO		———	"
CuO		———	"
PbO		———	"
ZnO		———	"
CaO		———	"
MgO		———	"
CO_2		———	"
SO_3		———	"
SiO_2, etc		———	"
Total		———	"

Where the lime and magnesia exist in amounts more than necessary to combine with the carbon dioxide and sulphur trioxide present, the excess may have united with oleic acid to form soaps insoluble in water, but soluble in ether.

In some instances the lead oxide and zinc oxide will be found only in the ether soap solution (3), as lead and zinc oleates, but in others, while they undoubtedly first existed as oleates, they had become decomposed, and the lead and zinc oxides would be found in section (8) of the above scheme.

The following is an analysis of a cylinder deposit, " Mica Grease" having been used as a lubricant :

Moisture		11.23	per cent.
Oils soluble in ether	Animal	9.05	"
	Mineral	6.28	"
Soap (CaO + MgO united with fatty acids)		43.90	"
Fixed carbon		6.33	"
Oxides of iron and aluminum		6.59	"
CaO		3.15	"
MgO		2.19	"
CO_2		6.27	"
Silica and mica		5.01	"
Total		100.00	"

References:—"The Production of Paraffin and Paraffin Oils." By R.H. Brunton, C. E., *Proc. Inst. Civ. Eng.*, **66**, 180–237.

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"The Manufacture of Paraffin Oil." By D. R. Stewart, F.C.S., *Ibid*, **8**, 100–110.

"Wool-Fat, and the Processes of Obtaining It." By H. W. Langbeck, *Ibid*, **9**, 356–359.

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"The Flashing Test for Petroleum." By F. A. Abel, F.R.S., *Ibid*, 1, 471–478.

"Report[1] on Lighting Passenger Equipment." Master Carbuilders' Association for 1893. *The Railway Car Journal*, July, 1894.

XLVIII.

Paint Analysis.

Paint is a liquid preparation having a two-fold use. Primarily it acts as a protecting coating against the action of the weather, and simultaneously as a decorative agent.

The liquid is usually linseed oil and turpentine and the coloring matter or body some solid pigment, such as finely ground red oxide of iron.

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[1] This report includes a list of the principal railroads in the United States, and the methods used by each for passenger car illumination; *viz.*, oil lamps, oil gas under pressure ("Pintsch gas"), or electric light (incandescent), with the conclusion that the "Pintsch gas" is rapidly being adopted in preference to oil illumination.

It is essential in the production of a good paint that the oil used should be one that, upon drying on the surface applied, should become hard, lustrous, and somewhat elastic.

Linseed oil excells all others in use for this purpose, and any sophistication thereto only deteriorates the quality.

Four qualities are essential in a paint: 1. Durability; 2. Working Qualities; 3. Drying Properties; 4. Covering Power.

The following list of pigments, with their chemical composition stated, will give an idea of the great variety that can be used in paints for outside work. The list would be largely increased were other pigments included that are used for interior decorative work only.

Red Pigments.—Indian red, Tuscan red (Fe_2O_3), vermilion (HgS), red lead (Pb_3O_4), antimony vermilion (Sb_2S_3). Iron oxide, Indian red, and Tuscan red can be analyzed by Scheme XIII, p. 29.

Brown Pigments.—Umbers (Fe_2O_3, MnO_2, etc.), Van Dyke brown, (Fe_2O_3, carbon), manganese brown (Mn_3O_4) and sepia. The composition of sepia is as follows:

Melanin	78.00	per cent.
$CaCO_3$	10.40	"
$MgCO_3$	7.00	"
Alkaline sulphates	2.16	"
Organic mucus	1.84	"
	99.40	"

White Pigments.—White lead ($2PbCO_3.PbH_2O_2$), lead sulphate ($PbSO_4$), zinc white (ZnO), sulphide of zinc, white (ZnS), "lithophone." Also the following, added oftentimes as fillers: barytes ($BaSO_4$), "blanc Fixe" (artificial barytes), gypsum ($CaSO_4$), strontium white ($SrSO_4$), whiting ($CaCO_3$), China clay (kaolin), and magnesite ($MgCO_3$).

Yellow and Orange Pigments.—Chrome yellow ($2PbCrO_4$), Chinese yellow ($PbO.PbCrO_4$), zinc chrome ($ZnCrO_4$), realgar (As_2S_2), "cadmium yellow" (CdS), "King's yellow" (As_2S_3), yellow ochre (Fe_2O_3, Al_2O_3, SiO_2, etc.), and Siennas (Fe_2O_3, H_2O, Mn_3O_4).

Green Pigments.—Chrome green (Cr_2O_3), copper green (CuA), mineral green (malachite), cobalt green (ZnO, CoO, P_2O_5, etc.), manganese green (BaO, MnO_2, etc.), emerald green ("Paris green," $7Cu2C_2H_3O_2$, $3CuAs_2O_4$) and Brunswick green (compounded of barytes, chrome yellow, Prussian blue, etc.).

Black Pigments.—Lampblack (carbon), bone-black (carbon and Ca_2HPO_4), vegetable black, Frankfort black, coal-tar black, asphaltum black, and graphite black (C).[1]

Blue Pigments.—Ultramarine (SiO_2, Al_2O_3, Na_2O, S), Prussian blue, Chinese blue, or Brunswick blue, ($Fe_7C_{18}N_{18}$), cobalt blue or smalts (Al_2O_3, CoO), Bremen blue (CuH_2O_2), and copper blue (CuO, CO_2, H_2O).

The various colored lakes, carmines, aniline lakes, etc., have but a limited application in Engineering Chemistry. Their methods of manufacture and assay can be advantageously studied by reference to "Painters' Colors, Oils, and Varnishes," by George H. Hurst, F.C.S., London, 1892, pp. 249–282.

The analysis of a white paint, ground in oil, as shown in the Scheme on page 455, will indicate the method of procedure in analyses of this character. Where qualitative analysis has shown the presence of a few constituents only, the Scheme can be correspondingly modified.

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[1] *The American Engineer and Railroad Journal*, Nov. 1896, p. 315, states: "Graphite mixed with an oil is chemically inert and in drying forms a coat that adheres firmly to the metal surface. Its resistance to the action of acids and alkalies has been proven by numerous tests much more severe than the conditions of service, and its resistance to the penetrations of moisture have been equally satisfactory. Heat does not cause it to blister, and we are informed that steel chimneys painted with it have been heated to redness without decolorizing the paint. The paint has been used with success upon the hulls and decks of steel steamers, and there seem to be no conditions of service which it does not successfully meet."

Scheme for the Analysis of White Paint Ground in Oil.

It may contain silica, **barium sulphate, lead sulphate, calcium sulphate, lead carbonate, clay, zinc oxide and** sulphide,[1] barium carbonate, calcium carbonate, linseed oil, rosin oil[2] and mineral oil.

Select a thin glass flask (150 cc.), weigh it and transfer to it about six grams of the paint and reweigh. Add fifty cc. petroleum ether, cork the flask, and set aside two hours. Pour the clear ether solution upon a filter, and collect filtrate in a weighed beaker. Repeat this operation until the last ether extract is colorless. Transfer residue to filter and wash with ether.

1. Residue may contain $PbSO_4$, $2PbCO_3PbH_2O_2$, $BaSO_4$, $CaSO_4$, $CaCO_3$, $BaCO_3$, ZnO, SiO_2, Clay, $ZnCO_3$, etc. Dry this residue and treat with hot acetic acid in a No. 2 beaker. Dilute with water, warm, and filter.

2. Filtrate contains the oils. Evaporate carefully to expel the ether and determine the weight of the oil.

If qualitative analysis has shown the presence of mineral oil, in addition to the linseed oil, proceed as follows:

Take 20 grams of the paint, exhaust with ether, and so obtain enough oil for saponification, etc.

After expulsion of the ether, add 100 cc. of an alcoholic solution of potash and evaporate until all the alcohol is expelled. Add 100 cc. water, dissolve any soap formed, and transfer all to a separatory funnel, add 50 cc. of sulphuric ether, shake well, and set aside twelve hours. The linseed oil, as soap, is drawn off in the water solution, while the mineral oil remains unsaponified in the ether solution.

Transfer this latter to a glass evaporating dish, expel the ether, and determine weight of mineral oil. This weight, subtracted from total weight of the two oils taken, gives the weight of the linseed oil.

3. Residue may contain SiO_2, clay, $PbSO_4$, $CaSO_4$, $BaSO_4$. Transfer the filter and contents to a small flask and digest, in the cold, for twelve hours with a saturated solution of $(NH_4)_2CO_3$. Filter, wash well with water. Neglect the filtrate. The residue on the filter is now treated with hot acetic acid; filter, wash well.

4. Filtrate may contain zinc, lead, barium, and calcium salts. Saturate with H_2S and filter, wash with H_2S water.

5. Residue may contain $BaSO_4$, SiO_2, or clay. Dry, ignite in platinum crucible and fuse with excess of Na_2CO_3; cool, treat with boiling water, filter, wash well with hot water.

6. Filtrate may contain $2PbC_2H_3O_2$, $Ca(C_2H_3O_2)_2$. Saturate with H_2S gas, filter.

7. Residue. $BaCO_3$. Dissolve in HCl, dilute with water, boil, add excess of dilute H_2SO_4, set aside 2 hours, filter, wash, dry, ignite and weigh as $BaSO_4$.

8. Filtrate. Acidify with HCl, evaporate to dryness in a porcelain evaporating dish. Take up with HCl, and water, boil, filter, and wash with hot water.

Residue. SiO_2. Dry, ignite and weigh as SiO_2.

Filtrate contains the Al_2O_3. Make alkaline with NH_4OH, boil, filter off the Al_2O_3, dry, ignite, weigh as Al_2O_3 and calculate to clay.

9. Residue contains PbS. Dry, transfer to a weighed porcelain crucible, add HNO_3, evaporate with H_2SO_4, dry, ignite, and weigh as $PbSO_4$.

10. Filtrate contains CaO. Boil out the H_2S, add NH_4OH, then $(NH_4)_2C_2O_4$, set aside two hours, filter, dry, ignite, and weigh as CaO, and calculate to $CaSO_4$.

11. Residue.—Pbs and Zns. Dissolve in HNO_3, add H_2SO_4, evaporate nearly to dryness, cool, add water, filter, wash well.

12. Filtrate.—Boil out H_2S, add HCl, and dilute H_2SO_4, to precipitate the barium. Filter, wash.

13. Residue.—Dry, ignite, and weigh as $PbSO_4$, and calculate to $2PbCO_3$ PbH_2O_2. ("Basic lead carbonate.")[3]

14. Filtrate.—Make alkaline with Na_2CO_3, boil, filter off the $ZnCO_3$, wash, dry, ignite, and weigh as ZnO. If H_2S has been indicated by qualitative analysis, calculate to ZnO, ZnS.

15. Residue.—Dry, ignite, and weigh as $BaSO_4$, and calculate to $BaCO_3$.

16. Filtrate Add NH_4OH to alkaline reaction, then $(NH_4)_2C_2O_4$. set aside two hours, filter, wash, dry ignite, and weigh as CaO and calculate to $CaCO_3$.

$BaSO_4$.	SiO_2.	$Al_2(SiO_3)_3$	$PbSO_4$.	$CaSO_4$.	$2PbCO_3$ PbH_2O_2.	ZnO, ZnS.	$BaCO_3$.	$CaCO_3$.	Linseed oil.	Mineral oil.

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[1] Zinc White is usually a mixture of ZnO and ZnS. "Lithophone," a zinc white, contains: barium sulphate, 67 per cent.; zinc oxide, 8 per cent.; zinc sulphide, 25 per cent.

[2] Rosin oil is best determined in a mixture of saponifiable and non-saponifiable oils, by the method of Twitchell. Consult *Journal of Analytical Chemistry*, 5, 379.

[3] For comparison of the different methods for determining the carbonic acid in paints consult "The Determination of Carbonic Acid in White Lead," by F. C. A. Meisel, *The School of Mines Quarterly*, 14, 318.

Analysis of White Lead Paints.[1]

(Dry, not ground in oil.)

	1.	2.	3.	4.	5.	6.
PbO	86.35	85.93	83.77	84.42	86.5	86.24
CO_2	10.44	11.89	15.06	14.45	11.3	11.68
H_2O	2.95	2.01	1.01	1.36	2.2	1.61
Total	99.74	99.83	99.84	100.23	100.0	99.53

from which the composition of the white leads can be calculated to be:

	1.	2.	3.	4.	5.	6.
$PbCO_3$	63.35	72.15	91.21	87.42	68.36	70.87
PbH_2O_2	36.14	27.68	8.21	12.33	31.64	28.66
Moisture	0.25	....	0.42	0.48	....	....
Total	99.74	99.83	99.84	100.23	100.00	99.53

No. 1. English make. Made by Dutch process; of very good quality.

No. 2. " " " " " " " " " "

No. 3. Krems white. Made by precipitation with carbon dioxide. It is deficient in body, although of good color.

No. 4. German make. Precipitated by carbon dioxide; of good color, but deficient in body.

No. 5. German make. Made by Dutch process; a good white.

No. 6. German make. Made by precipitation with carbon dioxide; quality fair.

Lead white, ground in oil, is a common form in the market. It usually contains about eight per cent. of raw linseed oil, and has an extended use among painters, as it readily mixes with additional oil and turpentine to form liquid paint.

The brown pigments, composed principally of oxides of iron and manganese, can be analyzed by Scheme XIII, p. 29; the yellows and greens containing chromium require a special process, as follows:

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[1] Painters' Colors, Oils and Varnishes. By G. H. Hurst, F.C.S., p. 30.

Scheme for the Analysis of Mixed Chromate and Sulphate of Lead (Lemon Chrome)[1] Not Ground in Oil.

Pulverize the sample, pass through a 100-mesh sieve, and mix. To one gram in a small beaker, add hydrochloric acid and heat. Any insoluble matter, (usually barytes as a gross adulteration) is to be filtered out, washed, ignited and weighed.

Lead.—One gram is treated in a covered casserole with twenty-five cc. concentrated sulphuric acid and heated moderately until the residue is perfectly white; cool, dilute with fifty cc. water and again cool; add fifty to seventy-five cc. of ninety-four per cent. alcohol, stir, and allow to stand one hour. Filter, wash well with alcohol, dry, ignite and weigh as $PbSO_4$.

Chromium and sulphuric acid (SO_3).—Treat one gram with about twenty-five cc. concentrated hydrochloric acid, boil, dilute to 100 cc. and while hot add excess of ammonium hydroxide, which precipitates the chromium and the greater part of the lead. Boil off the excess of ammonia, filter and wash *carefully* with hot water.

1. **Precipitate for Cr.**—Dissolve in dilute HCl, nearly neutralize acid with NH_4OH, precipitate Pb with H_2S gas and filter into a *porcelain dish*.

2. **Filtrate for SO_3.**—Acidify with HCl, concentrate, add boiling solution of $BaCl_2$ drop by drop and determine SO_3 as usual.

3. **Precipitate.**—PbS, reject.

4. **Filtrate.**—Boil off H_2S and precipitate Cr with NH_4OH in the usual manner. Put the moist precipitate and filter paper into a crucible and ignite carefully. Weigh as Cr_2O_3.

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[1] Analysis of Chrome Paints. By W. L. Brown. *J. Anal. Chem.*, 1, 213-215.

Occasionally the following determinations are made:

Water.—Hygroscopic. Heat one-half gram at 105° C. in an air-bath to constant weight.

Volatile Matter.—Heat one gram in a porcelain crucible to low redness; loss, less water, is volatile matter.

Water Extract.—(Acetates, sulphates, bichromates, or nitrates, indicating imperfect washing in manufacture.) Treat three grams with six successive portions of twenty-five cc. each, of cold distilled water, decanting and filtering each time, and evaporate the filtrate in a platinum dish to dryness on a water-bath.

Analysis of Mixed Chromate, Sulphate and Carbonate of Lead.

(Lemon, Chrome and White Lead.)

Analysis made same as in scheme for Lemon Chrome; excess of lead is to be calculated to white lead, $2PbCO_3+PbH_2O_2$.

Analysis of Red Chromate of Lead.[1]

For the lead determination take one gram in a covered casserole, add twenty-five cc. concentrated nitric acid, heat to boiling, and *while boiling* add half a dozen drops, one at a time, of alcohol, by means of a pipette; boil a while longer, add water, and all of the chromate, if it is pure, will be found in solution.

Without this alcohol treatment great difficulty will be experienced in getting the chromate into solution; with it, it becomes very easy. Add twenty-five cc. concentrated sulphuric acid, evaporate to white fumes and complete the analysis as described. For chromium and sulphur trioxide determinations, boil off alcohol and proceed as previously directed.

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[1] Known by various names, as scarlet, dark or basic chromate of lead, chrome red, Chinese red, American vermilion and vermilion substitute. Formula: $2PbO.CrO_3$ or $PbCrO_4 + PbO$.

Analysis of Chrome Green.

(Composed of Yellow Chromate of Lead, Prussian Blue and Lead Sulphate.)

To one gram of sample add twenty-five cc. HCl, heat to boiling several minutes, add water, allow to stand ten minutes, then filter and wash thoroughly with hot water.

1. **Residue.** — Prussian blue (plus barytes or other insoluble matter if present). Dry and ignite to Fe_2O_3.

Weight multiplied by 2.21 equals per cent. Prussian blue.

2. **Filtrate.**—Nearly neutralize with NH_4OH, leaving, however, the solution slightly acid. Pass H_2S gas through till Pb is all precipitated. Filter and wash.

3. **Precipitate,** PbS.—Dissolve on filter with hot dilute HNO_3 and boil the solution. Filter from collected S and bring filtrate of $Pb(NO_3)_2$ to small bulk, with several additions of H_2SO_4. Evaporate to fumes of H_2SO_4, cool, add water and alcohol, filter, wash and weigh as $PbSO_4$.

4. **Filtrate.**—For Cr (and Fe), boil off H_2S, add NH_4OH, in slight excess, boil this off and wash the $Cr_2(OH)_6$ (and $Fe_2(OH)_6$), as customary. Weigh precipitate as $Cr_2O_3 + Fe_2O_3$. After the weight is obtained, mix with one part KNO_3 and three parts Na_2CO_3; fuse in platinum crucible to clear fusion, cool, boil with water, filter and wash.

5. **Residue.**—Filter, wash, dry, and weigh as Fe_2O_3, if it is wanted as a check.

6. **Filtrate,** for Cr.—Precipitate with NH_4OH in glazed porcelain dish. If the weight of Cr_2O_3 is *very nearly* the same as before, then there has been no Fe extracted from the Prussian blue by the acid treatment. Some varieties are affected by this, others not. If the weight is less than the original, deduct it from same. The result is Fe_2O_3, which is also to be calculated to Prussian blue and added to the other.

Chrome green, in which the coloring matter is Cr_2O_3, is seldom found in the market pure. Usually it contains from twenty per cent. to seventy-five per cent. of barium sulphate.

As an example of specifications for a compound chrome paint, the following is given :

PENNSYLVANIA RAILROAD COMPANY. MOTIVE POWER DEPARTMENT.

Specifications for Cabin Car Color.

The standard cabin car color is the pigment known as scarlet lead chromate. It is always purchased dry. The material desired under this specification is the basic chromate of lead ($PbCrO_4PbO$), rendered brilliant by treatment with sulphuric acid and as free as possible from all other substances.

The theoretical composition of basic lead chromate is nearly 59.2 per cent. of the normal lead chromate, and 40.8 per cent. of lead oxide, but in the commercial article it is found that a portion of the sulphuric acid added to brighten the color remains in combination apparently with the normal lead chromate, slightly increasing the percentage of this constituent.

Samples showing standard shade will be furnished on application, and shipments must not be less brilliant than sample. The comparison of sample from shipment with the standard shade, may be made either dry or by mixing both samples with oil.

Shipments of cabin car color will not be accepted which

1. Contain barytes or any other adulterant.
2. Show on analysis less than fifty-seven per cent. or more than sixty per cent. of normal lead chromate, including the sulphuric acid combined as above stated.
3. Show on analysis less than thirty-eight per cent. or more than forty-two per cent. lead oxide, in addition to the lead oxide in the normal lead chromate.
4. Vary from standard shade.

Office of Gen. Supt. of Motive Power, Altoona, Pa., Feb. 18, 1891.

The various red paints, Indian red, Tuscan red, and other iron oxides, etc., used in general practice are rarely pure, but contain added amounts of finely pulverized gypsum and calcium carbonate. These oxides, when properly ground and mixed with linseed oil, form paints that cannot be excelled for durability, permanence of color and cheapness. Many of the mixtures contain varying amounts of japans, but as the japans have been subject to great sophistications of late years, specifications now generally call for linseed oil, turpentine and pigment only.

Thus, two varieties of paints might be roughly classified as: (1) Paints for wood work, and (2) paints for iron work.

The following specifications refer to class 1:

PENNSYLVANIA RAILROAD COMPANY. MOTIVE POWER DEPARTMENT.

Specifications for Freight Car Color.

Freight car color will be bought in the paste form, and the paste must contain nothing but oil, pigment and moisture.

The proportions of oil and pigment must be so nearly as possible as follows:

Pigment seventy-five per cent. by weight.

Oil twenty-five per cent. by weight.

The oil must be pure raw linseed oil, well clarified by settling and age. New process oil is preferred. The pigment desired contains not over one-half per cent. of hygroscopic moisture, and has the following composition:

Sesquioxide of iron, fifty per cent. by weight.

Fully hydrated calcium sulphate or gypsum, forty-five per cent. by weight.

Calcium carbonate, five per cent. by weight.

Samples of standard pigment showing shade will be furnished, and shipments will be required to conform with the standard.

The shade of paint being affected by the grinding, the Pennsylvania Railroad standard shade is that given by the dry sample sent, mixed with the proper amount of oil and ground, or better rubbed up in a small mortar with pestle until the paste will pass Pennsylvania Railroad test for fine grinding. It is best to use fresh samples of the dry pigment for each day's testing. The comparison should always be made with the fresh material, and never with the paint after it has become dry. The comparison is easiest made by putting a small hillock of the standard paste and of that to be compared near each other on glass, and then laying another piece of glass on the two hillocks, and pressing them together until the two samples unite. The line where the two samples unite is clearly marked if they are not the same shade. The paste must be so finely ground that when a sample of it is mixed with half its weight of pure raw linseed oil, and a small amount of the mixture placed on a piece of dry glass, there will be no separation of the oil from the pigment for at least half an hour. The temperature affects this test, and it should always be made at 70° F. The sample under test runs down the glass in a narrow stream when it is placed vertical, and it is sufficient if the oil and pigment do not separate for an inch down from the top of the test.

Shipments will not be accepted which

1. Contain less than twenty-three per cent. or more than twenty-seven per cent. of oil.
2. Contain more than two per cent. of volatile matter, the oil being

dried at 250° F., and the pigment dried in air not saturated with moisture at from 60° to 90° F.

3. Contain impure or boiled linseed oil.

4. Contain in the pigment calcium sulphate not fully hydrated, less than forty per cent. of sesquioxide of iron, less than two per cent. or more than five per cent. calcium carbonate, or have present any barytes, aniline colors, lakes, or any other organic coloring matter, or any caustic substances, or any makeweight or inert material which is less opaque than calcium sulphate.

5. Varying from shade.

6. Are not ground finely enough.

7. Are a "liver" or so stiff when received that they will not readily mix for spreading.

Altoona, Pa., Office Supt. Motive Power.

As an example of the composition of a paint for iron surfaces (Class 2) the following mixture as used for painting the structure of the elevated railroads in New York City is given:[1]

Boiled linseed oil	9	parts.
Red oxide of iron finely ground	7½	"
Turpentine	1	part.

In mixing paints for iron surfaces, it is of the first importance that only the best materials be used. Linseed oil is the best medium, when free from admixture with much turpentine.

The large percentage of linolein formed in drying, makes the surface of the paint solid and of a resinous appearance, possessing toughness and elasticity. Linseed oil does not crack or blister, by reason of the expansion and contraction of the iron with variation of temperature. Another important characteristic is its expansion while drying, which adapts it to iron surfaces. The Metropolitan Elevated Railway Company experimented very thoroughly with the various kinds and colors of paints; their labors at last culminated in the selection of a metallic paint for the first coat (formula given above) and a white lead paint for the second and last coat, both paints to contain the best linseed oil and enough turpentine to make the paints cover well and facilitate their drying.

The formula of the white lead paint as used is here given:

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[1] On the Construction of the Second Avenue Line of the Metropolitan Elevated Railway of New York. By G. Thomas Hall, C.E., *Trans. Am. Soc. Civil Eng.*, 10, 130.

WHITE LEAD PAINT. OLIVE COLOR.

147.42	kilograms	white lead.
79.38	"	" lime ($CaSO_4$).
34.02	"	French ochre.
1.36	"	Prussian blue.
0.45	"	burnt umber.
79.50	liters	boiled linseed oil.
5.67	"	turpentine.
3.79	"	liquid drier (boiled linseed oil and lead oxide).

Some engineers prefer red lead instead of iron oxide as the pigment for paints to be used for iron structures.

G. Bouscaren, C.E., states with regard to the painting of bridges, that having used both varieties of paint, he gives preference to the red lead.[1]

The red lead paint adheres better to the iron and fails principally by wear and a gradual transformation of the red lead into carbonate, whilst the iron paint fails by scaling.

Asphalt Paint.

Until within quite recent years little has been known in this country of the valuable properties of the asphalt. In the popular mind it is often confused with certain coal-tar products, which, though similar in appearance, differ essentially from asphalt in character. Asphalt oils are of a nearly non-volatile nature, and are therefore permanent, while on the other hand, coal-tar is volatile.

The so-called asphalt paints which have been used in the past are such only in name. They contain, at best, but a very small per cent. of asphalt, which is incorporated in the form of a pigment and which serves no valuable purpose. Asphalt, on the contrary, should be the main constituent, since the value of such a paint depends upon the presence of the permanent asphalt oils.[2]

Fire Proof Paints, Silicate Paints, Asbestos Paints, etc.

The principle of action of these paints is not to render wood work or similar material fire proof, but to retard combustion.

Wood treated with a solution of zinc chloride. or with a solu-

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[1] Trans. Amer. Soc. Civil Engineers, 15, 429.

[2] *Am. Eng. and R. R. Journal*, 65, 185.

tion of sodium silicate, can be rendered nearly non-inflammable, and after such treatment and drying, paint can be applied.

Instead of using the ordinary paints for this purpose, various compounds are incorporated in the paint itself to render the latter non-inflammable. Thus the preparation of Prof. Abel J. Martin, of Paris, is as follows:

Boracic acid, borax, soluble cream of tartar, ammonium sulphate, potassium oxalate, and glycerine mixed with glue and incorporated with a paint. It is the result obtained after many experiments in response to a prize of 1000 francs, offered by the Society for the Advancement of National Industry of France. A committee consisting of Professors Dumas, Palaird and Troost, after testing the materials, consisting of painted woods and various fabrics, for seven months, reported in favor of this preparation. The municipality of Paris made its use obligatory in all of the theatres there and it has stood the test of the last six years.

Blue Pigments. Ultramarine beinga silicate, can be analyzed by Scheme XIV, page 37.

COMPOSITION OF ULTRAMARINE.

SiO_2	49·68	per cent.
Al_2O_3	23·00	"
S	9·23	"
SO_3	2·46	"
Na_2O	12·50	"
H_2O	3·13	"
Total	100·00	"

COMPOSITION OF COMMERCIAL PRUSSIAN BLUE.

Al_2O_3	2·45	per cent.
Fe_2O_3	3·31	"
$CaSO_4 + SiO_2$	89·86	"
$CN + S$	4·11	"
H_2O	0·27	"
Total	100·00	"

COMPOSITION OF SMALTS.

SiO_2	56·4	per cent.
Al_2O_3	3·5	"
Fe_2O_3	4·1	"
CoO	16·0	"
CaO	1·6	"
K_2O	13·2	"
PbO	4·7	"
Total	99·5	"

Examination of the Oil after Extraction from the Paint.

The only adulterants used in linseed oil, in this connection, are mineral oil and rosin oil. Their method of detection and estimation is given on page 414.

Turpentine, when present, is not an adulterant, and a mixture, extracted from a paint, may contain linseed oil, mineral oil, rosin oil, turpentine, and rosin spirit.[1] The latter is quite distinct from rosin oil and when properly prepared is a perfect substitute for turpentine. If the liquid extracted from the paint is a mixture of linseed oil, turpentine, and rosin spirit the determination of the amounts of each is somewhat difficult.

Turpentine can be distinguished and determined in the presence of rosin spirit by the action of the former on polarized light, rosin spirit being inert. Thus: the specific rotation of American turpentine varies between +8.8 to +21.5. The bromine absorption is also an indication:

The bromine absorption of turpentine varies between 203 and 236.

The bromine absorption of rosin spirit varies between 184 and 200.

The determination of the amounts of petroleum naphtha and turpentine in a mixture can be made by the method of H. E. Armstrong, *J. Soc. Chem. Ind.*, **1**, 480; consult also Allen, Com. Org. Anal., **2**, 48–50.

References: "How to Design a Paint." By C. B. Dudley, *Railway and Eng. J.*, **65**, 174, 318.

"On the Analysis of White Paint." By G. W. Thompson, *J. Soc. Chem. Ind.*, **15**, 432.

"Detection of Rosin and Rosin Oil in Oils and Varnishes." By F. Ulzer, *Ibid*, **15**, 382.

"Technical Analysis of Asphaltum." By L. A. Linton, *J. Am. Chem. Soc.*, **16**, 809.

"Rustless Coatings for Iron and Steel, Galvanizing, Electro-Chemical Treatment, Painting and Other Preservative Methods." By M. P. Wood, *Trans. Am. Soc. Mec. Eng.*, **16**, *1895*, 350–450.

"Preservative Coatings for Iron Work." By A. H. Sabin and A. O. Powell, *Engineering News*, Feb. 5, 1895, p. 86.

"Chemische Operationen der Analyse von Farbstoffen." By F. Schmidt, Mitth. Malerei, **9**, 121.

"Chemistry of Paints and Painting." By A. H. Church. London, 1892.

"Pigments, Paints and Painting." By G. Terry. London, 1893.

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[1] Coal tar naphtha has an extended use in the preparation of varnishes: the use of it in paints, however, is very limited.

XLIX.

Pyrometry.

Pyrometry, or the art of measuring high temperatures, has received, in the past few years, considerable attention from engineers and metallurgists.

This is especially so in the direction of metallurgical engineering, where more uniform methods of heating and controlling heat have developed. In many processes of melting, refining, tempering, etc., certain temperatures are required, from which, should much variation occur, the products would be ruined.

Many forms of pyrometers have been invented, but only a very few have accomplished their purpose. Many are admirable in design and construction, and prove accurate and trustworthy in the laboratory, but fail utterly when applied in practice at high temperatures.

Pyrometers may be classified according to the principles upon which they operate:[1]

1. Expansion of mercury in a glass tube. When the space above the mercury is filled with compressed nitrogen and a specially hard glass is used for the tube, mercury thermometers can indicate correct temperatures to 1,000° F.
2. Contraction of clay, as the Wedgewood pyrometers; very inaccurate as the contraction of the clay varies with the composition of the clay.
3. Expansion of air, as in the air thermometer, Siegert's pyrometer, Wiborgh's pyrometer, Uehling & Steinbart's pyrometer, etc.
4. Pressure of vapors, as the Spannung's pyrometer, or the Bristol recording thermometer.
5. Relative expansion of two metals, as Brown's or Buckley's pyrometers.
6. Specific heat of solids, as iron-ball, copper-ball, or platinum-ball pyrometer.
7. Melting-point of metals, alloys, etc.
8. Time required to heat a weighed quantity of water—a water pyrometer.

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[1] *Engineering News*, 34, 322 (Nov. 14, 1895).

9. Increase in temperature of a stream of water or other liquid flowing at a given rate through a tube inserted into the heated chamber, as the Saintignon pyrometer.

10. Changes in the electric resistance of platinum or other metal, as in the Siemen's pyrometer.

11. Measurement of an electric current produced by heating the junction of two metals, as in the Le Chatelier pyrometer.

12. Dilution of a stream of hot air or gas flowing from a heated chamber by cold air, and determination of the temperature of the mixture by a mercury thermometer, as in Hobson's hot-blast pyrometer.

13. Polarization and refraction, by prisms and plates, of light radiated from heated surfaces, as in Mesure and Noel's optical pyrometer.

The standard of reference for all temperatures above 212° F. is the air thermometer and all pyrometers are usually standardized by comparison with it.

The various forms of air thermometers are described by Prof. R. C. Carpenter in *Engineering News*, Jan. 5, 1893.

The air pyrometer of Messrs. A. Siegert and W. Duerr, Fig. 146, consists of a porcelain cylinder connected by a thin copper tube with the measuring portion of the apparatus. This consists of a bell of sheet brass, the lower edge of which dips into a bath of petroleum.

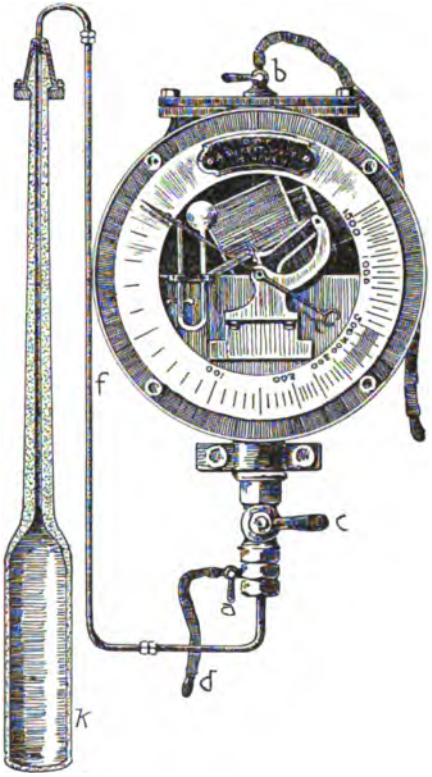

Fig. 146.

The bell is attached to one arm of a balance beam, a counterpoise being carried by the other arm.

The porcelain bulb being heated to the temperature to be measured, the air it contains expanding enters the brass bell, lifting this and moving the beam. The movement is shown on a scale and the temperature read direct from the divisions, into which the scale is divided. (Consult *Jour. Iron and Steel Inst.*, 1893, p. 340.

Wiborgh's air pyrometer is fully described in *Trans. Am. Inst. Mining Eng.*, **21**, p. 592.

Hobson's hot-blast pyrometer is largely used for measuring the temperature of the blast in hot-blast iron furnaces. It consists of a brass chamber having three arms and a handle, Fig. 147.

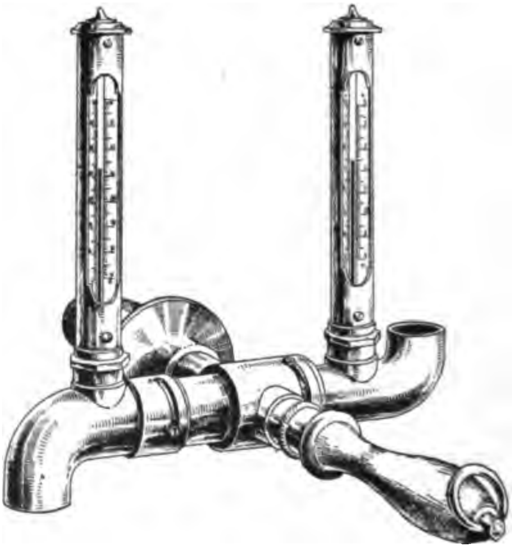

Fig. 147.

An opening through a jet in one of the arms admits the hot blast, another arm admits atmospheric air, while the third arm is for the discharge of the mixture. To this third arm is attached a thermometer which indicates the temperature of the mixed current. A thermometer is also attached to the arm admitting the atmospheric air.

This pyrometer can be used constantly to 2000° F., without danger of injury.

Bristol's recording thermometer gives a continuous graphical record of temperatures up to 600° F. It consists of a copper coil which takes the place of a bulb and is inserted in the heated space. The bulb is partly filled with alcohol, which is partly vaporized by the heat. The pressure of the vapor is transmitted through a fine flexible copper tube, filled with alcohol, to any convenient distance not exceeding twenty-five feet, where it is measured by a recording pressure gauge. The interior of the gauge contains a flat Bourdon spring coiled into three complete coils. The movable end of the spring carries a pointer, which contains an inking pencil at its outer end. The clock-work revolves a paper chart once in twenty-four hours, and the marker thus makes a continuous record.

The metallic pyrometer of Brown or Bulkley's form consists of the well-known copper and iron tube, and is based on the principle of the difference of expansion between copper and iron. An iron tube is encased loosely in a copper tube, the two being connected at one end. At the other end the exterior tube is connected to the casing of a graduated dial and the inner tube to a multiplying gear, which multiplies the relative motion of the free ends of the tubes and moves a rotating pointer on the dial. Temperature, higher than 1500° F., cannot be accurately measured with this instrument.

The Copper-Ball or Platinum-Ball Pyrometer. If a weighed piece of metal, such as iron, copper, or platinum, be allowed to remain in a furnace or heated chamber till it acquires the temperature of the chamber and then be suddenly taken out and immersed in a vessel containing a quantity of water of known weight and temperature, the resulting increased temperature of the water may be used as a measure of the temperature of the ball when it was withdrawn from the furnace.

A modification of the Weinhold pyrometer by Schneider is shown in Fig. 148.

The calorimeter proper *g*, is surrounded by the containing vessel *m*, of sheet lead; the space between *g* and *m* is filled with air but conduction at *p* is reduced by a layer of paste-board. The cover *d* admits the thermometer *t*, the upright rod *w*, connected with the paddler *r*, is kept in motion by speed imparted to the wheel *v*. In practice the heated ball *k* is dropped through *a*, at the same instant *c* closes, and *k* falls into the wire net *l*. After thorough agitation of the water by *r*, the maximum rise of temperature of the water is taken.

Let W = the weight of the water, w = weight of the ball, t = the original and T the final temperature of the water, and S the

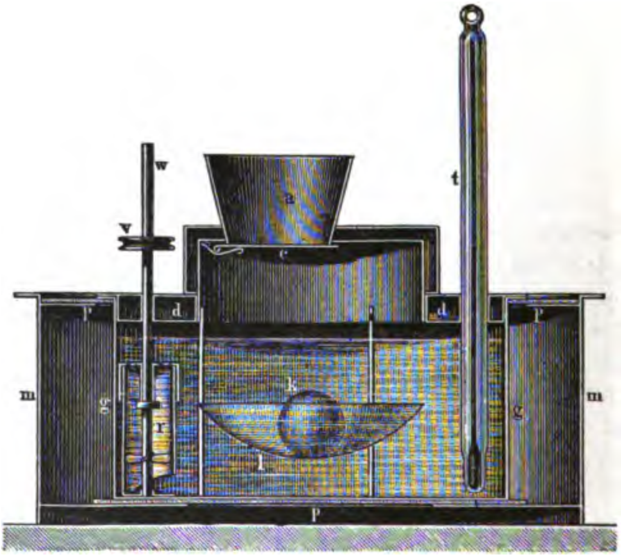

Fig. 148.

specific heat of the metal, then the temperature of the heated chamber may be found from the following formula :

$$x = W\left(\frac{T-t}{w\,S}\right) + T$$

In practice many precautions are required. The metal ball should be enclosed in a small crucible, or other casing while in the furnace and until the instant the ball is dropped into the water, in order to avoid loss by radiation during the transfer from the furnace to the water; the water should be stirred rapidly in order to cool the ball as quickly as possible; the "water equivalent" of the heat-carrying capacity of the vessel containing the water should be carefully determined and added to the actual quantity of water used, to obtain the corrected value of W in the formula. Finally for scientific determinations, the actual specific heat of the metal ball should be carefully determined. The specific heat of metals generally increases with the temperature; thus the specific heat of wrought iron, according to Petit and Dulong is 0.1098 from 32° to 42° F., and 0.1255 from 32° to 662° F. The specific heat of copper is 0.094 from 32° to 212° F., and 0.1013 from 32° to 572° F. The mean specific heat of platinum between 32° and 446° F. is 0.0333, and it increases 0.0003 for each increase of 100° F. For complete instructions regarding the use of the platinum ball for determining high temperatures consult *Trans. Am. Soc. Mech. Eng.*, 6, 702.

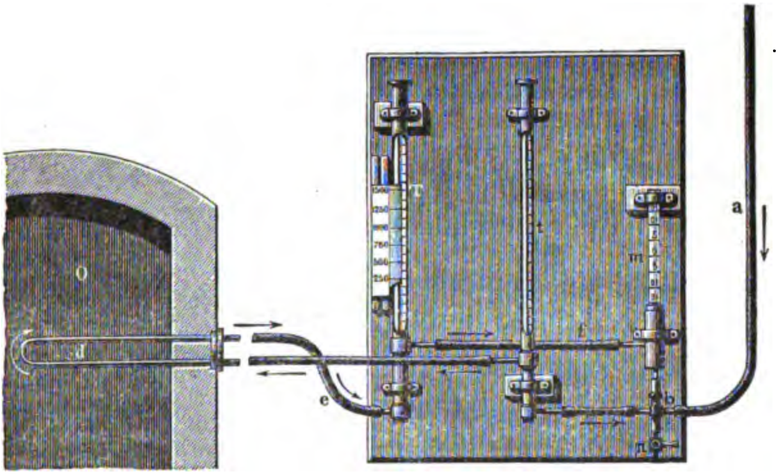

Fig. 149.

The use of pyrometers dependent upon the melting point of alloys is extremely limited. The Seger Fire Clay Pyrometer which is included in this classification is fully described by H. M. Howe, in the *Engineering and Mining Journal*, June 7, 1890.

A pyrometer dependent upon increase of the temperature of a stream of water flowing through a tube in the heated chamber is shown in the Saintignon pyrometer Fig. 149.

Through the tube *a* enters a regulated stream of water, the temperature of which is measured by the thermometer *t*. The water passes through the heated oven *o* by means of the copper tube *d* and the increase of temperature is indicated by the thermometer *T;* from thence by the tube *f*, to the manometer *m* and then through *n* it leaves the pyrometer.

A water-current pyrometer invented by Carnelly and Burton is fully described in "Grove and Thorp's Chemical Technology," 1, 342.

Of the electrical pyrometers, four have had an extended use; *viz;* Siemen's pyrometer, the electric pyrometer of Prof. Braun, the thermo-electric pyrometer of Le Chatelier, and the Simond's thermo-electric pyrometer. A description of the Siemen's electric pyrometer will be found in *Proceedings Royal Soc.*, 1886, p. 566.

This pyrometer has been superseded by the Le Chatelier pyrometer.

Prof. Braun's Electric Pyrometer.

The principle of its action is based upon the electrical resistance of platinum wire when exposed to high temperatures. The platinum wire is in a long fire-proof tube and is wound upon a fire-clay cylinder free from induction. It forms a part of a Wheatstone's Bridge which in connection with a sensitive galvanometer permits the resistance to be measured rapidly and conveniently and the corresponding temperature is directly obtained. The measuring apparatus proper is contained in a box (Fig. 150) so constructed that only the parts to be handled are visible, while the battery is placed in a separate compartment. The necessary manipulations are very simple.

After the pyrometer has been placed in the heated chamber and the connection made with the measuring apparatus, the lever in the latter is turned forward to close up the circuit with the batteries and galvanometer. Then the graduated arc must be so placed that the pointer of the galvanometer (Fig. 151) is at zero, when the index on the arc (Fig. 150) will indicate at once the temperature of the pyrometer in degrees centigrade.

The distance between the pyrometer and the measuring apparatus may be quite considerable, twenty-five to thirty feet.

Measurements are considered accurate up to 1500° C.

Le Chatelier's Thermo-Electric Pyrometer.

When wires of two dissimilar metals or alloys are placed in contact with each other and highly heated at the point of contact, an electric current is generated, the strength of which varies with the temperature, and may be indicated by a galvanometer. The pyrometer consists of a thermo-electric couple of two wires, one of pure platinum and the other of platinum alloyed with ten per cent. of rhodium and are connected with a D'Arsonaal galvanometer.

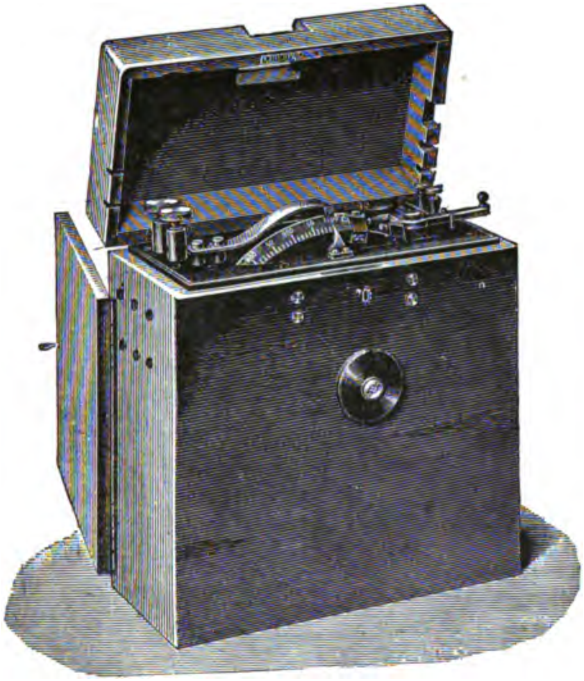

Fig. 150.

The couple is inserted into the furnace or oven whose temperature is to be measured, and the current is led by wires to the galvanometer placed at any convenient distance from the couple.

The instrument is capable of measuring very high temperatures.

A complete description of this pyrometer, by H. M. Howe, is given in *Trans. Am. Inst. Min. Eng.*, 24, 746.

The Mesure and Nouel pyrometric telescope is fully described in the *Engineering and Mining Journal*, June 7, 1890.

The Uehling and Steinbart pneumatic pyrometer represents the latest advances in pyrometry and is having an extended use in iron blast furnace work.

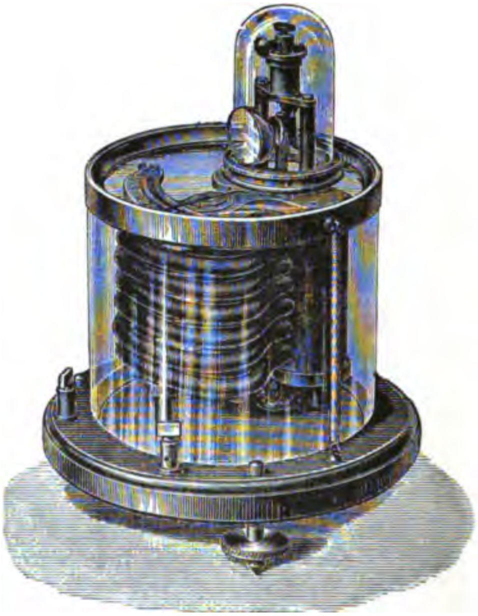

Fig. 151.

This instrument Figs. 153, 154, 155, is designed especially for continuously indicating high temperatures, for making an autographic record of the heat conditions, and is based on the laws governing the flow of air through small apertures. If two such apertures *A* and *B*, Fig. 152, respectively form the inlet and outlet openings of a chamber *C*, and a uniform suction is created in the chamber *C'* by the aspirator *D*, the action will be as follows: Air will be drawn through the aperture *B* into the chamber *C'*, creating suction in chamber *C*, which in turn causes air from the atmosphere to flow in through the aperture *A*. The velocity with which the air enters through *A* depends on the suction in the chamber *C*, and the velocity at which it flows out through *B* depends upon the excess of suction in *C'* over that existing in the chamber *C*, that is, ***the effective suction*** in *C'*. As the suction in *C* increases, the effective suction must decrease, and hence the velocity at which air flows in through the aperture *A* increases and the velocity at which air flows out through the aperture *B* decreases, until the same quantity of air enters at *A* as passes out at *B*. As soon as this occurs no further change of suction can take place in the chamber *C*.

Air is very materially expanded by heat. Therefore the higher the temperature of the air the greater the volume, and the smaller will be the quantity of air drawn through a given aperture by the same suction. Now if the air, as it passes through the aperture *A* is heated, but again cooled to a lower fixed temperature before it passes through the aperture *B*, less air will enter through the aperture *A* than is drawn out through the aperture *B*. Hence the suction in *C* must increase and the effective suction in *C*, must decrease, and in consequence the velocity of the air through *A* will increase and the velocity of the air through *B* will decrease, until the same quantity of air again flows through both apertures.

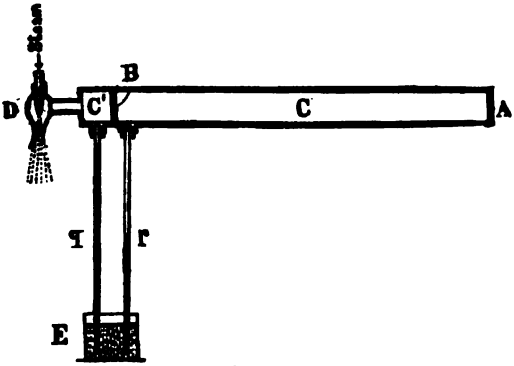

Fig. 152.

Thus every change of temperature in the air entering through the aperture *A* will cause a corresponding change of suction in the chamber *C*.

If two manometer tubes *p* and *q*, Fig. 152, communicate

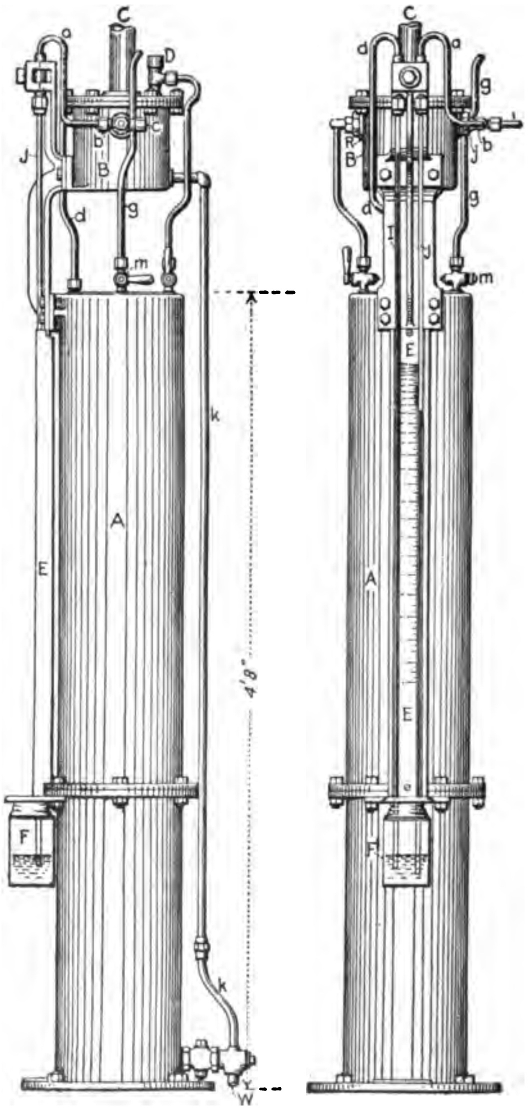

Fig. 153.

respectively with the chambers *C* and *C'* the column in tube *q* will indicate the constant suction in *C'* and the column in tube *p* will indicate the suction in the chamber *C*, which suction is a true measure of the temperature of air entering through the aperture *A*.

This principle was very fully demonstrated previously by Prof. Barus, *in U. S. Geol. Survey, Bulletin No. 54*, 1889, p. 239.

Practical application of the above principles is made in the pneumatic pyrometer of Messrs. Uehling and Steinbart. Fig. 153 shows a side and front elevation of the instrument, and Fig. 154 shows the fire tube in connection with a hot-blast main of a blast furnace, and also a filter, *K*, for purifying the air to prevent the obstruction of the small apertures by particles of dust, etc. The fire tube *M*, Fig. 154, projects into the heated chamber. The air enters by *h* into the fire tube *M*, in which is to be heated to the temperature to be measured, and at this temperature it enters the small aperture at the end of the inner tube *i j* into a coil, located in chamber *B* (Fig. 153), thence through the second aperture, located in the coupler *R*, into the air space above the water in the vessel *A*, from which it is continuously drawn by the aspirator *D*.

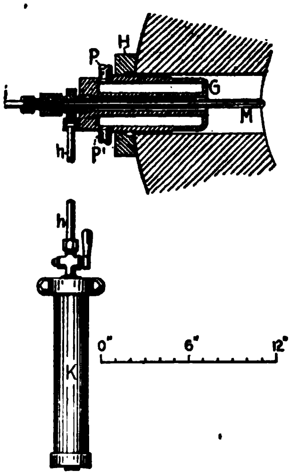

Fig. 154.

A pipe, open at both ends, enters the vessel *A* from the top and dips into the water exactly forty-eight inches. The aspirator consists of a platinum tube, closed at one end, and having placed concentrically within it a smaller platinum tube, which has a small aperture at its end. The connection of the fire tube with the other pipes, which are of drawn copper, is protected against injury from the heat by the cooler *G*, held in position by the flange *H*, and is provided with water circulation by means of the pipes *P P'*. The vessel *A*, four feet eight inches in height and eight inches internal diameter, and filled with water to within six inches of the top, serves as a suction regulator, and the vessel *B*, into which the exhaust steam of the aspirator *D* is discharged, serves as the temperature regulator. Two manometer tubes, *I* and *J*, are fastened in front of the scale *E*, and dip into the liquid contained in the jar *F*.

The tube *I* connects through the pipe *d* with the top of the regulator *A*, and shows the amount of suction, as at *I'*. The tube *J* connects by the tube *a* at *b* with the space between the two small apertures, one of which is located in the end of the inner fire-tube, and the other in the coupling, *R*, just within the vessel *B*. A water connection is provided, by which the vessel *A* may be filled to the proper level.

The instruments operate as follows: Steam being turned on the aspirator *D*, a partial vacuum is at once created in the apparatus. In consequence, atmospheric air enters the bottom of the tube *K* (Fig. 154), which tube being filled with cotton, cleanses the air from all dust, etc., and allows it to pass through the connecting tube apertures, the deficiency being drawn through the tube just described against the constant water column of forty-eight inches. This insures a perfect and automatic regulation of the suction, which is always shown by the manometer *I* at *I'*.

If the water column in *A*, in consequence of the gradual evaporation, sinks, it will at once show at *I'*, and can be replenished by simply opening the cock at *M* until *I'* comes to the exact mark.

The aspirator *D* exhausts into the vessel *B*, and from there through *C* into the atmosphere; the water of condensation drains off by the pipe *K* into the waste pipe *W*. By this expedient the temperature in *B* is constantly kept at 212° F., and as the air passes through a coil located in *B*, it must assume this temperature before passing through the second aperture.

Having thus secured, first, a constant difference of tension of the air before entering the first aperture and after leaving the second aperture, and also a constant temperature at which it passes through the second aperture, the tension between the two apertures must necessarily vary with the temperature of the air entering through the first aperture, which is located at the end of *n*. The manometer, *J*, communicating with the tube or chamber between the two apertures through the pipe *a*, indicates the temperature surrounding the fire-tube, and can be read off on the scale *EE* at *J'*, for example.

The connecting pipe, *i*, may be several hundred feet longer, so that the main instrument, Fig. 153, can be placed in a convenient place a considerable distance away from the hot-blast main furnace, etc., the temperature of which is to be measured.

This pyrometer records correctly the temperature as high as 2,500° F. and in many instances at 2,700° F.

Prof. W. C. Roberts-Austen gives, as the results of many determinations by various pyrometers, the following boiling and melting-points:

Melting-point of lead	326° C.
Boiling-point of mercury	358° "
Melting-point of zinc	415° "
Boiling-point of sulphur	448° "
Melting-point of aluminum	625° "
Boiling-point of selenium	665° "
Melting-point of silver	945° "
" " " gold	1045° "
" " " copper	1054° "
" " " palladium	1500° "
" " " platinum	1775° "

References:—"The Thermal Limit." By E. H. Griffiths. *Phil. Mag.*, **40**, 431. (Capacity for heat of water at different temperatures. Consideration of certain thermal units other than those dependent on the capacity for heat of water.)

"On the Determination of High Temperatures by Means of Platinum Resistance Pyrometers." By C. T. Heycock and F. H. Neville, *J. Chem. Soc.*, 1895, p. 160.

"Ueber die Messung hoher Temperaturaten. By L. Holborn and W. Wein, *Pogg. Annalen*, N. F., **56**, p. 360. (Die Oefen, Prüfung der Constanz der Thermo-elemente, Schmelzpunkte von Nickel, Palladium, Platin, Widerstandsänderung von Platin- und Palladiumdrähten unter dem Einfluss von Wasserstoff und Kieselsäure, Widerstandsänderung von reinem Platin und Rhodium mit der Temperatur, Luftthermometergefässe aus schwerschmelzbarer Masse.)

"Sur un Thermomètre a Zéro Invariable." M. L. Marchis, *J. d. Phys.*, **4**, p. 217.

"The Thermophone." By C. Warren Whipple, *Electric*, **36**, 285.

"Pyrometry and the Heat Treatment of Steel." By Henry M. Howe, *Trans. Am. Inst. Min. Eng.*, **24**, p. 746.

"Recent Advances in Pyrometry." By W. C. Roberts-Austen, F.R.S., *Trans. Am. Inst. Min. Eng.*, **24**, pp. 407–444.

L.

The Electrical Units.

The electrical units may be derived from the three fundamental units of length, mass. and time, and so defined are known as the centimeter-gram-second units; or, in short, the C. G. S. units. These units are as follows:

Centimeter = unit of length.
Gram = unit of mass.
Second = unit of time.
Dyne = unit of force, equal to that force which acting on one gram for one second, produces a velocity of one centimeter per second.
Erg. = unit of work, equal to the work done by one dyne acting through the distance of one centimeter.

These are, in general, either too large or too small for practical purposes, so that the practical units are taken as multiples or fractions of C. G. S. units.

Two distinct systems may be derived, the electrostatic system, having for its basis the repulsion of two like charges of electricity, and the electromagnetic system, having for its basis the repulsion of two like magnetic poles. Only the latter system need be here considered. In this system the *Unit Magnetic Pole* is that which repels an equal and similar pole at one centimeter distance with a force of one dyne. Unit pole produces unit magnetic field at a distance of one centimeter from it. *Unit current* is one which, in a wire of one centimeter length, bent into an arc of one centimeter radius, would act upon a unit pole placed at the center with a force of one dyne.

Practical Units.

These were adopted by the International Electrical Congress, Chicago, 1893, and are generally known as the international units.

Current.—The *Ampère* is one-tenth of the C. G. S. unit of current; practically represented by that current which, under standard conditions, deposits silver at the rate of 0.001118 gram per second.

An ordinary 50-volt incandescent lamp takes a current of about one ampère; an arc lamp requires about ten ampères.

Resistance.—The *Ohm* is the resistance of an uniform column of pure mercury 106.3 centimeters long and 14.4521 grams in mass, at 0° C.

The cross section of this column is one square mm. 100 feet of No. 20 B. and S. copper wire have an approximate resistance of one ohm, at the ordinary temperature.

Electromotive Force.—The *Volt* is the E. M. F., which steadily applied to a conductor whose resistance is one ohm, will produce in it a current of one ampère.

It is practically represented by $\frac{1000}{1434}$ part of the E. M. F. of a Clark standard cell at 15° C.

A Daniel cell has an E. M. F. slightly greater than one volt.

Quantity.—The *Coulomb* is the quantity of electricity conveyed by one ampère in one second.

Capacity.—The *Farad* is that capacity which requires one coulomb of electricity to charge it to a potential of one volt. For ordinary use, the one-millionth part, or micro-farad, is employed as the unit.

Work.—The *Joule* is the energy expended in one second by one ampère in one ohm. It is equal to 107 ergs. Expressed in heat units, one joule = 0.24 calories. (Calorie = gram-degree at 4° C.)

Power.—The *Watt* is the power expended by one ampère flowing under a pressure of one volt; it is equal to work done at the rate of one joule per second. 746 watts are approximately equal to one horse power.

Inductance.—The *Henry* is such a disposition of the circuit that a change of current at the uniform rate of one ampère per second induces a counter-electromotive force of one volt.

For convenience of expression, quantities respectively one million times greater or smaller than these are sometimes designated by the prefixes *megra-* and *micro-*. Thus insolation resistances are usually expressed in *megohms*, one megohm being equal to one million ohms; capacites, in terms of *microfarads*, a microfarad being equal to the one-millionth part of a farad. The prefixes *kilo-* and *milli-* denote quantities respectively one thousand times greater or smaller than the units to which they are prefixed.

Thus dynamo machinery is ordinarily rated in ***kilo-watts***, one kilo-watt being equal to one thousand watts, or very nearly equal to one and one-third horse power; small currents, such as are used in medicine, are frequently expressed in milli-ampères.

The relations between the international units of resistance and electromotive force, to those of the older units, are:

1 B. A. unit = 0.98660 International unit.
1 International unit = 1.01358 B. A. units.
1 Legal unit = 0.99718 International unit.
1 International unit = 1.00283 Legal unit.

OHM'S LAW.—The current flowing in any *complete* circuit is equal to the total electromotive force, divided by the total resistance of the circuit. For any *part* of a circuit, *not containing a source of E. M. F.*, the current flowing is equal to the difference of potential between the ends of the part, divided by the resistance of that part.

So, in general,
$$\text{Ampères} = \frac{\text{Volts}}{\text{Ohms}}.$$

JOULE'S LAW.—The heat developed in any conductor is proportional;

1st, to its resistance,

2nd, to the square of the current strength,

3rd, to the time that the current lasts.

The quantitative relation of these, known as Joule's Law, is
$$U = 0.24 \times C^2Rt,$$
or in units
$$\text{Calories} = 0.24\ (\text{Ampères})^2 \times \text{Ohms} \times \text{Seconds}.$$

Measurement of Electric Energy.—The electrical energy given to any part of a circuit can be found by placing an ampère-meter in series with the circuit, and a volt-meter in shunt with the circuit.

The product of ampères and volts gives the Watts and this divided by 746 gives the horse-power.

That is
$$\text{Horse-power} = \frac{\text{Ampères} \times \text{Volts}}{746}.$$

The ampère-meter and volt-meter may be combined into one instrument, called a watt-meter, which gives the power directly in watts.

ELECTRO-CHEMICAL EQUIVALENTS.

	Grams per coulomb.
Hydrogen	0.000010334
Gold	0.0006791
Silver	0.0011180
Copper (Cupric)	0.000328
Mercury (Mercuric)	0.0010374
Zinc	0.0033698
Oxygen	0.00008286
Water	0.00009315

LI.

Energy Equivalents.

There frequently occur, in the course of engineering work, calculations of efficiency and consumption which are, more or less, long and tedious. The figures given in the following paragraphs will reduce any such calculation to a case of simple multiplication or division. This not only saves time, but greatly decreases the chance of errors, which can often pass unnoticed in many of the rarely understood and complicated expressions which such calculations involve. Only full theoretical values or equivalents are given, and when the delivery is not up to the figure the deficiency is the loss in the transformation, or if the consumption is greater than the equivalent, such excess is the waste of the process. Some of the equivalents are, at the present time, uncertain, and the figures given are subject to such changes as their definite determination will involve. Joule's equivalent has been used as 776, which is considered a conservative figure, as is also the light equivalent of 1 C. P. = 620 foot-pounds per hour. Logarithms of each number have been inserted, and the reciprocal of any equivalent will be found under its proper heading.

WORK.

One (1) Horse-Power =

In Foot-Pounds. 33,000 (log. 4.518514) foot-pounds per minute.
550 (log. 2.740363) foot-pounds per second.
1,980,000 (log. 6.296665) foot-pounds per hour.

In B. T. U.	.709 (log. $\bar{1}.850646$) B. T. U. per second.
	42.53 (log. 1.628652) B. T. U. per minute.
	2,552 (log. 3.406710) B. T. U. per hour.
In Pounds Steam.	2.219 (log. 0.346105) pounds of steam per hour at 80 pounds pressure (95 pounds absolute).
	2.2104 (log. 0.344441) pounds steam at 100 pounds pressure (115 pounds absolute).
In Combustion.	.002933 (log. $\bar{3}.467312$) pounds carbon consumed per minute, or 0.176 (1.24551) pounds carbon per hour.
	.1823 (log. $\bar{1}.260787$) pounds ordinary coal per hour.
	.1169 (log. $\bar{1}.067815$) pounds = 0.0157 gals. (log. $\bar{2}.19590$) ordinary petroleum per hour.
	.1276 (log. $\bar{1}.105781$) pounds good kerosene per hour
	3.925 (log. 0.593890) cubic feet ordinary house gas per hour.
In Electricity and Light.	746 (log. 2.872739) watts or
	2,750 (log. 3.43933) candle power.

One (1) Foot-Pound per Second =

In H. P.	.001818 (log. $\bar{3}.259594$) horse power.
In Electric Light.	1.3565 (log. 0.132343) watts, or
	5 (log. 0.698970) candles.
In B. T. U.	4.64 (log. 0.666515) B. T. U. per hour.
In Steam.	.004034 (log. $\bar{3}.605699$) pounds steam at 80 pounds pressure (95 pounds absolute) per hour.
	.004018 (log. $\bar{3}.604035$) pounds steam at 100 pounds pressure (115 pounds absolute) per hour.

One Foot-Pound per Minute =

In H. P.	.0000303 (log. $\bar{5}.481443$) H. P.
In Electric Light.	.0226 (log. $\bar{2}.354108$) watts.
	.0833 (log. $\bar{2}.920820$) candles.
In B. T. U.	.07733 (log. $\bar{2}.888348$) B. T. U. per hour.
In Steam.	.00006723 (log. $\bar{5}.827548$) pounds steam at 80 pounds pressure (95 pounds absolute) per hour.
	.00006696 (log. $\bar{5}.825874$) pounds steam at 100 pounds pressure (115 pounds absolute) per hour.

In Rotary Delivery.

Rotary Delivery to Get H. P.	A force of 52.41 (log. 1.719333) pounds at an arm 1 foot long, making 100 revolutions per minute, gives one H. P.
	A force of 100 pounds acting on an arm 1 foot long, making 52.41 (log. 1.719333) revolutions per minute, gives 1 H. P.

A force of 100 pounds, acting on an arm 0.5241 (log. $\bar{1}.619333$) foot = 6¼ inches long, making 100 revolutions per minute, gives 1 H. P.

A force of 100 pounds, acting on an arm 1 foot long, and making 100 revolutions per minute, gives 1.904 (log. 0.279665) H. P.

Roughly we have 1 H. P. for 100 pounds pull on a belt running over a 1-foot pulley (1 foot diameter), making 100 revolutions per minute.

HEAT.

One B. T. U. (1 Pound Water Raised 1° F.) =

776 (log. 2.889862) foot pounds.

One B. T. U. Consumed per Second =

B. T. U. to Work, Light and Electricity.

1.411 (log. 0.149500 horse power, or

1,052.6 (log. 3.022263) watts, or

3,880 (log. 3.588832) candle power.

One B. T. U. per Minute =

.023515 (log. $\bar{2}.371345$) H. P., or

17.5433 (log. 1.244112) watts, or

64.66 (log. 1.810569) candles.

One B. T. U. per Hour =

.000392 (log. $\bar{4}.593200$) H. P., or

.2924 (log. $\bar{1}.465977$) watts, or

1.078 (log. 0.032619) candles.

One Pound of Steam.

Steam to Work, Light and Electricity.

At 100 pounds pressure (115 absolute) takes .7962 (log. $\bar{1}.901000$) pounds carbon, or 0.0824 (log. $\bar{2}.915927$) pounds ordinary good coal to make it from water at 62^{c} F., assuming no loss; it contains

1,154.5 (log. 3.062368) B. T. U., or

895,892 (log. 5.959315) foot-pounds.

If it were consumed in one hour it would represent—with no loss—

14,931 (log. 4.174089) foot-pounds per minute, or

.45247 (log. $\bar{1}.655565$) H. P., or

337.6 (log. 2.528304) watts, or

1,244.5 (log. 3.094893) candles.

One Pound of Steam.

Steam to Work, Light and Electricity.

At 80 pounds pressure (95 absolute) takes
.0793 (log. $\bar{2}.899328$) pounds carbon, or
.0821 (log. $\bar{2}.914343$) pounds ordinary good coal to make it from water at 62° F., assuming no loss. It contains
1,150 (log. 3.060698) B. T. U., or
892,400 (log. 5.950551) foot-pounds.
If it be consumed in 1 hour with no loss=
14,873 (log. 4.172400) foot-pounds per minute, or
.4507 (log. $\bar{1}.65388$) H. P., or
336.2 (log. 2.526625) watts, or
1239 (log. 3.09322) candles.

One Pound of Carbon Consumed in 1 Hour =

Combustion.

14,500 (log. 4.161368) B. T. U. per hour.
11,252,000 (log. 7.051230) foot-pounds per hour.
5.683 (log. 0.754565) H. P.
4,240 (log. 3.627304) watts.
15,630 (log. 4.193895) candles.

Fuels to B. T. U.

15 (log. 1.176091) pounds water evaporated from and at 212° F.
12.56 (log. 1.099000) pounds steam made from water at 62° F., to steam at 100 pounds pressure (115 pounds absolute).

Steam work.

12.61 (log. 1.10067) pounds steam made from water at 62° F. to steam at 80 pounds pressure (95 pounds absolute).

One Pound Ordinary Kerosene Consumed per Hour =

Light and Electricity.

20,000 (log. 4.301030) B. T. U. per hour.
15,520,000 (log. 7.190892) foot-pounds per hour.
7.838 (log. 0.894227) H. P.
5,847 (log. 3.766966) watts.
21,560 (log. 4.333557) candles.
20.7 (log. 1.316053) pounds water evaporated from and at 212° F.
17.325 (log. 1.238673) pounds water from 62° F. to steam at 100 pounds pressure (115 pounds absolute).
17.40 (log. 1.240050 pounds water at 625 F. to 80 pounds pressure (95 pounds absolute).

One Cubic Foot Ordinary Illuminating Gas per Hour =

650 (log. 2.812913) B. T. U. per hour.
504,400 (log. 5.702775) foot-pounds per hour.

.25475 (log. $\bar{1}$.406110) H. P.
190 (log. 2.278849) watts.
700 (log. 2.845440) candle power.
.6729 (log. $\bar{1}$.827936) pounds water evaporated from and at 212° F.
.563 (log. $\bar{1}$.750585) pounds water at 62° F. to steam at 100 pounds pressure (115 pounds absolute).

LIGHT.

One Candle Power =

Light to Work. .00036364 (log. $\bar{4}$.560672) H. P.
.2713 (log. $\bar{1}$.433411) watts.
12 (log. 1.079181) foot-pounds per minute.
720 (log. 2.857332) foot-pounds per hour.
B. T. U. .015464 (log. $\bar{2}$.189319) B. T. U. per minute.
Electricity, .92783 (log. $\bar{1}$.967470) B. T. U. per hour.
Steam and Combustibles. .0008037 (log. $\bar{4}$.905102) pounds steam per hour at 100 pounds pressure (115 pounds absolute).
.0008068 (log. $\bar{4}$.906772) pounds steam at 80 pounds pressure (95 pounds absolute).
.000064 (log. $\bar{5}$.806102) pounds.
.448 (log. $\bar{1}$.6512) grains carbon per hour.
.0000661 (log. $\bar{5}$.820201) pounds ordinary coal per hour.
.0000464 (log. $\bar{5}$.66644) pounds.
.32475 (log. $\bar{1}$.511538) grains.
.001531 (log. $\bar{3}$.184975) cubic inches.
0.000006628 (log. $\bar{6}$.821342) gallons ordinary kerosene per hour.
.001427 (log. $\bar{3}$.154557) cubic feet ordinary gas per hour.

ELECTRICITY.

One (1) Watt =

Electricity to Work. .0013405 (log. $\bar{3}$.127241) H. P.
.057 (log. $\bar{2}$.755913) B. T. U. per minute.
B. T. U. 3.42 (log. 0.534064) B. T. U. per hour.
Steam, 44.24 (log. 1.645775) foot-pounds per minute.
Light and 2,654.4 (log. 3.423966) foot-pounds per hour.
Combustibles. 3.6863 (log. .566591) candle power.
.000236 (log. $\bar{4}$.372696) pounds.
1.65 (log. .217794) grains carbon per hour.
.000171 (log. $\bar{4}$.233034) pounds.
1.197 (log. 0.078132) grains good kerosene per hour.
.005262 (log. $\bar{3}$.721151) cubic feet ordinary illuminating gas per hour.

LIST OF THE PRINCIPAL ELEMENTS, WITH THEIR ATOMIC WEIGHTS, SPECIFIC GRAVITIES AND SPECIFIC HEATS.

	Atomic weight.	Specific gravity.	Specific heat.
Aluminum	27.50	2.67	0.2143
Antimony	120.0	6.70	0.0508
Arsenic	75.0	5.63	0.0814
Barium	137.0	4.00	0.0470
Bismuth	208.0	7.67–9.93	0.0380
Boron	11.0	2.68	0.3660
Bromine	80.0	3.15	0.0843
Cadmium	112.0	8.45	0.0567
Calcium	40.0	1.58	0.1670
Carbon	12.0	2.33–3.52	0.4590
Chlorine	35.5	1.38 (liquid)	0.1800
Chromium	52.5	7.01	0.1000
Cobalt	59.0	8.957	0.1070
Copper	63.5	8.952	0.0950
Fluorine	19.0	...	0.2600
Gold	197.0	19.50	0.0324
Hydrogen	1.0	0.0692 (air = 1.0)	2.3000
Iodine	127.0	4.94	0.0541
Iridium	193.0	22.42	0.0326
Iron	56.0	7.79	0.1138
Lead	207.0	11.35	0.0306
Magnesium	24.0	1.70	0.2499
Manganese	55.0	8.03	0.1217
Mercury	200.0	13.60	0.0319
Molybdenum	96.0	8.56	0.0722
Nickel	58.8	9.50	0.1082
Nitrogen	14.0	0.971 (air = 1.0)	0.3600
Oxygen	16.0	1.105 (air = 1.0)	0.2500
Palladium	106.5	11.40	0.0593
Phosphorus	31.0	1.84	0.1895
Platinum	195.0	21.15	0.0324
Potassium	39.0	0.86	0.1655
Silicon	28.0	2.49	0.2030
Silver	108.0	10.53	0.0560
Sodium	23.0	0.98	0.2934
Strontium	87.5	2.542	0.1740
Sulphur	32.0	2.07	0.1776
Tin	118.0	7.20	0.0562
Titanium	48.0	3.588	0.1300
Uranium	240.0	18.40	0.0279
Vanadium	51.2	5.50	.....
Wolfram (tungsten)	184.0	18.3	0.0334
Zinc	65.0	7.37	0.0955

CONVERSION TABLES.

Found.	Sought.	Factor.	Found.	Sought.	Factor.
Al_2O_3	Al_2	0.53015	$Mg_2P_2O_7$	2Mg	0.21883
NH_4Cl	NH_3	0.31882	Mn_2O_3	2Mn	0.69695
$PtCl_6(NH_4)_2$	$2NH_3$	0.07692	Mn_3O_4	3Mn	0.72084
$PtCl_6(NH_4)_2$	N_2	0.06329	MnS	Mn	0.63211
Pt	$2NH_3$	0.17518	Hg	HgO	1.07984
$(NH_4)_2SO_4$	$2NH_3$	0.25815	HgS	Hg	0.86208
Sb_2O_3	Sb_2	0.83366	MoS	Mo	0.49992
Sb_2O_5	Sb_2	0.75046	NiO	Ni	0.78524
Sb_2S_3	Sb_2	0.71438	$NiSO_4$	Ni	0.37849
As_2O_3	As_2	0.75757	$(NH_4)_2PtCl_6$	2N	0.06329
As_2O_5	As_2	0.65217	$PbSO_4$	Pb	0.68292
As_2S_3	As_2	0.60928	Pt	2N	0.14414
$BaSO_4$	BaO	0.65654	PdI_2	Pd	0.29448
$BaSO_4$	Ba	0.58790	$Mg_2P_2O_7$	2P	0.27852
Bi_2O_3	2Bi	0.89654	$Mg_2P_2O_7$	P_2O_5	0.63756
$KBFl_4$	B	0.08683	U_2PtO_{11}	P_2O_5	0.19817
AgBr	Br	0.42556	$(NH_4)_2PtCl_6$	Pt	0.43911
CdS	Cd	0.77712	K_2SO_4	K_2	0.44898
$CdSO_4$	Cd	0.53786	K_2SO_4	K_2O	0.54075
CaO	Ca	0.71428	K_2PtCl_6	K_2O	0.19404
$CaSO_4$	CaO	0.41158	AgCl	Ag	0.75275
CO_2	C	0.27278	SiO_2	Si	0.47020
$CaCO_3$	CO_2	0.44002	$SiFl_4$	Si	0.57878
$BaCO_3$	CO_2	0.22332	Na_2SO_4	Na_2	0.32435
AgCl	Cl	0.24725	Na_2SO_4	Na_2O	0.43674
Cr_2O_3	Cr_2	0.68483	NaCl	Na	0.39408
Cr_2O_3	$2CrO_3$	1.31520	$BaSO_4$	S	0.13755
CoO	Co	0.78696	$BaSO_4$	SO_3	0.34346
CuO	Cu	0.79858	$SrSO_4$	Sr	0.47674
Cu_2S	Cu_2	0.79827	Tl_2PtCl_6	2Tl	0.50046
$CaFl_2$	Fl_2	0.48088	SnO_2	Sn	0.78681
$BaSiFl_6$	6Fl	0.40783	TiO_2	Ti	0.60065
AgI	I	0.54031	U_3O_8	3U	0.84873
Fe_2O_3	Fe_2	0.70000	Vd_2O_5	2Vd	0.56145
Fe_2O_3	2FeO	0.89999	WoO_3	Wo	0.79310
$LiCO_3$	Li_2	0.18944	ZnO	Zn	0.80338
MgO	Mg	0.60375	ZrO_2[1]	Zr	0.73913

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[1] Improvements in Methods of Chemical Calculations." Consult *J. Anal. Chem.*, 1, 402.

COMPARISON OF CENTIGRADE AND FAHRENHEIT DEGREES.

Degrees Centigrade.	Degrees Fahrenheit.	Degrees Centigrade.	Degrees Fahrenheit.	Degrees Centigrade.	Degrees Fahrenheit.
2500	4532	274	525.2	220	428
2000	3632	273	523.4	219	426.2
1500	2732	272	521.6	218	424.4
1200	1992	271	519.8	217	422.6
1000	1832	270	518	216	420.8
950	1742	269	516.2	215	419
900	1652	268	514.4	214	417.2
850	1562	267	512.6	213	415.4
825	1517	266	510.8	212	413.6
800	1472	265	509	211	411.8
775	1427	264	507.2	210	410
750	1382	263	505.4	209	408.2
725	1337	262	503.6	208	406.4
700	1292	261	501.8	207	404.6
675	1247	260	500	206	402.8
650	1202	259	498.2	205	401
625	1157	258	496.4	204	399.2
600	1112	257	494.6	203	397.4
575	1067	256	492.8	202	395.6
550	1022	255	491	201	393.8
500	932	254	489.2	200	392
475	887	253	487.4	199	390.2
450	842	252	485.6	198	388.4
425	797	251	483.8	197	386.6
400	752	250	482	196	384.8
375	707	249	480.2	195	383
350	662	248	478.4	194	381.2
325	617	247	476.6	193	379.4
300	572	246	474.8	192	377.6
299	570.2	245	473	191	375.8
298	568.4	244	471.2	190	374
297	566.6	243	469.4	189	372.2
296	564.8	242	467.6	188	370.4
295	563	241	465.8	187	368.6
294	561.2	240	464	186	366.8
293	559.4	239	462.2	185	365
292	557.6	238	460.4	184	363.2
291	555.8	237	458.6	183	361.4
290	554	236	456.8	182	359.6
289	552.2	235	455	181	357.8
288	550.4	234	453.2	180	356
287	548.6	233	451.4	179	354.2
286	546.8	232	449.6	178	352.4
285	545	231	447.8	177	350.6
284	543	230	446	176	348.8
283	541.4	229	444.2	175	347
282	539.6	228	442.4	174	345.2
281	537.8	227	440.6	173	343.4
280	536	226	438.8	172	341.6
279	534.2	225	437	171	339.8
278	532.4	224	435.2	170	338
277	530.6	223	433.4	169	336.2
276	528.8	222	431.6	168	334.4
275	527	221	429.8	167	332.6

Comparison of Centigrade and Fahrenheit Degrees—*Continued.*

Degrees Centigrade.	Degrees Fahrenheit.	Degrees Centigrade.	Degrees Fahrenheit.	Degrees Centigrade.	Degrees Fahrenheit.
166	330.8	112	233.6	58	136.4
165	329	111	231.8	57	134.6
164	327	110	230	56	132.8
163	325.4	109	228.2	55	131
162	323.6	108	226.4	54	129.2
161	321.8	107	224.6	53	127.5
160	320	106	222.8	52	125.6
159	318.2	105	221	51	123.8
158	316.4	104	219.2	50	122
157	314.6	103	217.4	49	120.2
156	312.8	102	215.6	48	118.4
155	311	101	213.8	47	116.6
154	309.2	100	212	46	114.8
153	307.4	99	210.2	45	113
152	305.6	98	208.4	44	111.2
151	303.8	97	206.6	43	109.4
150	302	96	204.8	42	107.6
149	300.2	95	203	41	105.8
148	298.4	94	201.2	40	104
147	296.6	93	199.4	39	102.2
146	294.8	92	197.4	38	100.4
145	293	91	195.8	37	98.6
144	291.2	90	194	36	96.8
143	289.4	89	192.2	35	95
142	287.6	88	190.4	34	93.2
141	285.8	87	188.6	33	91.4
140	284	86	186.8	32	89.6
139	282.2	85	185	31	87.8
138	280.4	84	183.2	30	86
137	278.6	84	181.4	29	84.2
136	276.8	82	179.6	28	82.4
135	275	81	177.8	27	80.6
134	273.2	80	176	26	78.8
133	271.4	79	174.2	25	77
132	269.6	78	172.4	24	75.2
131	267.8	77	170.6	23	73.4
130	266	76	168.8	22	71.6
129	264	75	167	21	69.8
128	262.4	74	165.2	20	68
127	260.6	73	163.4	19	66.2
126	258.8	72	161.6	18	64.4
125	257	71	19.58	17	62.6
124	255.2	70	158	16	60.8
123	253.4	69	156.2	15	59
122	251.6	68	154.4	14	57.2
121	249.8	67	152.6	13	55.4
120	248	66	150.8	12	53.6
119	246.2	65	149	11	51.8
118	244.4	64	147.2	10	50
117	242.6	63	145.4	9	48.2
116	240.8	62	143.6	8	46.4
115	239	61	141.8	7	44.6
114	237.2	60	140	6	42.8
113	23.54	59	138.2	5	41

COMPARISON OF CENTIGRADE AND FAHRENHEIT DEGREES—*Continued.*

Degrees Centigrade.	Degrees Fahrenheit.	Degrees Centigrade.	Degrees Fahrenheit.	Degrees Centigrade.	Degrees Fahrenheit.
4	39.2	— 8	17.6	—20	— 4
3	37.4	— 9	15.8	—21	— 5.8
2	3.56	—10	14	—22	— 7.6
+ 1	33.8	—11	12.2	—23	— 9.4
0	32	—12	10.4	—24	—11.2
— 1	30.2	—13	8.6	—25	—13
— 2	28.4	—14	6.8	—30	—22
— 3	26.6	—15	5	—35	—31
— 4	24.8	—16	3.2	—38	—36.4
— 5	23	—17	+ 1·4	—40	—40
— 6	21.2	—18	— 0.4		
— 7	19.4	—19	— 2.2		

STEAM PRESSURES EXPRESSED IN POUNDS PER SQUARE INCH AND ATMOSPHERES FOR DIFFERENT TEMPERATURES.

Pounds per square inch.	Atmospheres.	Temperature of steam.	Pounds per square inch.	Atmospheres.	Temperature of steam.
1	0.07		33	2.24	
2	0.14		34	2.31	
3	0.21	60° C.	35	2.38	
4	0.28	[140° F.]	36	2.45	
5	0.35		37	2.52	128.8° C.
6	0.41		38	2.58	[263.° F.]
7	0.48		39	2.65	
8	0.54		40	2.72	
9	0.61	86° C.	41	2.79	
10	0.68	[186.8° F.	42	2.86	
11	0.75		43	2.92	
12	0.81		44	2.99	135.1° C.
13	0.88		45	3.06	[275° F.]
14	0.95		46	3.13	
15	1.02	100° C.	47	3.20	
16	1.09	[212° F.]	48	3.26	
17	1.16		49	3.33	
18	1.23		50	3.40	
19	1.30		51	3.47	140.6° C.
20	1.36		52	3.54	[284° F.]
21	1.43		53	3.60	
22	1.50	112.2° C.	54	3.67	
23	1.56	[234° F.]	55	3.74	
24	1.63		56	3.81	
25	1.70		57	3.88	
26	1.77		58	3.94	145.4° C.
27	1.84		59	4.01	[294° F.]
28	1.90		60	4.08	
29	1.97		61	4.15	
30	2.04	121.4° C.	62	4.22	
31	2.11	[250.° F.]	63	4.28	
32	2.18		64	4.35	

STEAM PRESSURES EXPRESSED IN POUNDS PER SQUARE INCH AND ATMOSPHERES FOR DIFFERENT TEMPERATURES—*Continued.*

Pounds per square inch.	Atmospheres.	Temperature of steam.	Pounds per square inch.	Atmospheres.	Temperature of steam.
65	4.42		95	6.46	
66	4.49	149.1° C.	96	6.53	163.5° C.
67	4.56	[300.4° F.]	97	6.60	[325.3° F.]
68	4.62		98	6.66	
69	4.69		99	6.73	
70	4.76		100	6.80	
71	4.83		101	6.87	
72	4.95		102	6.94	
73	4.96		103	7.00	166.5° C.
74	5.03	153.1° C.	104	7.07	[331.7° F.]
75	5.15	[307.6° F.]	105	7.14	
76	5.17		106	7.21	
77	5.24		107	7.28	
78	5.35		108	7.35	
79	5.37		109	7.42	
80	5.44		110	7.49	169° C.
81	5.51	156.8° C.	120	8.17	[336.2° F.]
82	5.57	[314.2° F.]	130	8.85	
83	5.64		140	9.53	180° C.
84	5 71		150	10.21	[356° F.]
85	5.78		160	10.89	
86	5.85		170	11.57	190° C.
87	5.92		180	12.25	[374° F.]
88	5.98	160.2° C.	190	12.93	
89	6.05	[320° F.]	200	13.61	
90	6.12		210	14.29	
91	6.19		220	14.97	200° C.
92	6.25		230	15.65	[392° F.]
93	6.32		240	16.33	
94	6.39		250	17.01	257° C.
					[494.6° F.]

United States System of Measures and Weights Compared With the Metric System.

1. Linear Measure.

1 mile=8 furlongs=80 chains=320 perches=5280 feet=1609.344 meters.
1 furlong =10 chains= 40 perches= 660 feet= 201.168 "
1 chain = 4 perches= 66 feet= 20.1168 "
1 perch = 16½ feet= 5.0292 "
1 chain = 100 links.
1 link=7.92 inches=0.201168 meters.
1 yard=3 feet=36 inches=0.9144 "
1 foot=12 inches=0.3048 "
1 inch =0.0254 "

2. Surface Measures.

1 square mile=640 acres.
1 acre=10 square chains=160 square perches=43560 sq. feet=40.4694 ares.

3. Measures of Capacity.

A.—DRY MEASURE.

1 bushel=2150.42 cubic inches.
1 bushel=the volume of 77.627 pounds of distilled water at 4° C.
Legal: 1 liter=0.908 quart.
1 bushel=4 pecks=8 gallons=32 quarts=35.24229 liters.
1 peck =2 gallons= 8 quarts= 8.81057 liters.
1 gallon = 4 quarts= 4.40528 liters.
1 quart = 1.10132 liters.
1 cubic foot=7.48 gallons=28.315 liters=62.42 pounds of water at 60° F.

B—LIQUID MEASURE.

1 gallon=231 cubic inches.
1 gallon=the volume of 8.3388822 pounds=58378 troy grains of distilled water at 4° C.
Legal: 1 liter=1.0567 quart=0.26417 gallon.
1 gallon=4 quarts=8 pints=32 gills=3.78544 liters.
1 quart =2 pints= 8 gills=0.94636 liter.
1 pint = 4 gills=1.47318 liter.
1 gill =0.118295 liter.

4. Weights.

1 grain troy=0.0648004 gram.
1 pound troy=0.822857 pound avoirdupois.
1 pound avoirdupois=7000 grains troy=1.215279 pounds troy.

A—AVOIRDUPOIS WEIGHTS.

1 ton = 20 hundred weight = 2240 pounds = 1016.070 kilograms.
1 hundred weight = 112 pounds = 50.8035 kilograms.

1 pound = 16 ounces = 256 drams = 768 scruples = 7680 grains = 453.603 grams
1 ounce = 16 drams = 48 scruples = 480 grains = 28.350 grams
1 dram = 3 scruples = 30 grains = 1.772 grams
1 scruple = 10 grains = 0.5906 gram

B—TROY WEIGHT FOR DRUGS.

1 pound = 12 oz. = 96 drachms = 288 scruples = 5760 grains = 373.2503 gms.
1 oz. = 8 drachms = 24 scruples = 480 grains = 31.1042 gms.
1 drachm = 3 scruples = 60 grains = 3.888025 gms.
1 scruple = 20 grains = 1.296008 gms.
1 grain = 0.064804 gm.

C—TROY WEIGHT FOR JEWELS AND PRECIOUS METALS.

1 pound = 12 ounces = 24 carats = 240 pwts = 5760 grains = 373.2503 gms.
1 ounce = 2 carats = 20 pwts = 480 grains = 31.1042 gms.
1 carat = 10 pwts = 240 grains = 15.5521 gms.
1 pennyweight = 24 grains = 1.55521 gms.
1 grain = 0.0648004 gm.

Percentages and Gravity of Ammonia.

TABLE SHOWING THE PERCENTAGES OF AMMONIA (NH_3) IN AQUEOUS SOLUTIONS OF THE GAS OF VARIOUS SPECIFIC GRAVITIES.

Carius. Temperature 15° C.

Specific gravity.	NH_3 per cent.	Specific gravity.	NH_3 per cent.	Specific gravity.	NH_3 per cent.
0.8844	36	0.9133	24	0.9520	12
0.8864	35	0.9162	23	0.9556	11
0.8885	34	0.9191	22	0.9593	10
0.8907	33	0.9221	21	0.9631	9
0.8929	32	0.9251	20	0.9670	8
0.8953	31	0.9283	19	0.9709	7
0.8976	30	0.9314	18	0.9749	6
0.9001	29	0.9347	17	0.9790	5
0.9026	28	0.9380	16	0.9831	4
0.9052	27	0.9414	15	0.9873	3
0.9078	26	0.9449	14	0.9915	2
0.9106	25	0.9484	13	0.9959	1

Table Showing the Amount of K_2O in Potash Lye of Different Specific Gravities. Temperature 17.5°.

(Hoffman-Schaedler, "Tabellen für Chemiker," p. 119.)

K_2O per cent.	Specific gravity.	K_2O per cent.	Specific gravity.	K_2O per cent.	Specific gravity.	K_2O per cent.	Specific gravity.
45.0	1.576	34.0	1.414	23.0	1.269	12.0	1.135
44.5	1.568	33.5	1.407	22.5	1.263	11.5	1.129
44.0	1.560	33.0	1.400	22.0	1.257	11.0	1.123
43.5	1.553	32.5	1.393	21.5	1.250	10.5	1.117
43.0	1.545	32.0	1.386	21.0	1.244	10.0	1.111
42.5	1.537	31.5	1.379	20.5	1.238	9.5	1.105
42.0	1.530	31.0	1.372	20.0	1.231	9.0	1.099
41.5	1.522	30.5	1.365	19.5	1.225	8.5	1.094
41.0	1.514	30.0	1.358	19.0	1.219	8.0	1.088
40.5	1.507	29.5	1.352	18.5	1.213	7.5	1.082
40.0	1.500	29.0	1.345	18.0	1.207	7.0	1.076
39.5	1.492	28.5	1.339	17.5	1.201	6.5	1.070
39.0	1.484	28.0	1.332	17.0	1.195	6.0	1.065
38.5	1.477	27.5	1.326	16.5	1.189	5.5	1.059
38.0	1.470	27.0	1.320	16.0	1.183	5.0	1.054
37.5	1.463	26.5	1.313	15.5	1.177	4.5	1.048
37.0	1.456	26.0	1.307	15.0	1.171	4.0	1.042
36.5	1.449	25.5	1.301	14.5	1.165	3.5	1.037
36.0	1.442	25.0	1.294	14.0	1.159	3.0	1.031
35.5	1.435	24.5	1.288	13.5	1.153	2.5	1.026
35.0	1.428	24.0	1.282	13.0	1.147	2.0	1.021
34.5	1.421	23.5	1.275	12.5	1.141	1.5	1.015

Table Showing the Amount of Sodium Oxide (Na_2O) in Soda Lyes of Different Specific Gravities. Temperature 17.5°.

(Hoffman-Schaedler, "Tabellen für Chemiker.")

Na_2O per cent.	Specific gravity.	Na_2O. per cent.	Specific gravity.	Na_2O per cent.	Specific gravity.	Na_2O. per cent.	Specific gravity.
35.0	1.500	27.5	1.389	20.0	1.281	12.5	1.174
34.5	1.492	27.0	1.382	19.5	1.274	12.0	1.167
34.0	1.485	26.5	1.375	19.0	1.266	11.5	1.160
33.5	1.477	26.0	1.367	18.5	1.259	11.0	1.153
33.0	1.470	25.5	1.360	18.0	1.252	10.5	1.146
32.5	1.463	25.0	1.353	17.5	1.245	10.0	1.139
32.0	1.455	24.5	1.345	17.0	1.238	9.5	1.132
31.5	1.448	24.0	1.338	16.5	1.231	9.0	1.125
31.0	1.440	23.5	1.331	16.0	1.224	8.5	1.118
30.5	1.433	23.0	1.324	15.5	1.217	8.0	1.111
30.0	1.426	22.5	1.317	15.0	1.210	7.5	1.104
29.5	1.418	22.0	1.309	14.5	1.203	7.0	1.097
29.0	1.411	21.5	1.302	14.0	1.195	6.5	1.090
28.5	1.404	21.0	1.295	13.5	1.188	6.0	1.083
28.0	1.396	20.5	1.288	13.0	1.181	5.5	1.076

Specific Gravity of Solutions of Calcium Chloride at 18.3° (Schiff.)

Specific gravity.	Per cent. $CaCl_2+6H_2O$.	Per cent. $CaCl_2$.	Specific gravity.	Per cent. $CaCl_2+6H_2O$.	Per cent. $CaCl_2$.
1.0039	1	0.507	1.1575	36	18.245
1.0079	2	1.014	1.1662	37	18.752
1.0119	3	1.521	1.1671	38	19.259
1.0159	4	2.028	1.1719	39	19.766
1.0200	5	2.534	1.1768	40	20.272
1.0241	6	3.041	1.1816	41	20.779
1.0282	7	3.548	1.1865	42	21.286
1.0323	8	4.055	1.1914	43	21.793
1.0365	9	4.562	1.1963	44	22.300
1.0407	10	5.068	1.2012	45	22.806
1.0449	11	5.575	1.2062	46	23.313
1.0491	12	6.082	1.2112	47	23.820
1.0534	13	6.587	1.2162	48	24.327
1.0577	14	7.096	1.2212	49	24.834
1.0619	15	7.601	1.2262	50	25.340
1.0663	16	8.107	1.2312	51	25.847
1.0706	17	8.611	1.2363	52	26.354
1.0750	18	9.121	1.2414	53	26.861
1.0794	19	9.625	1.2465	54	27.368
1 0838	20	10.136	1.2516	55	27.874
1.0882	21	10.643	1.2567	56	28.381
1.0927	22	11.150	1.2618	57	28.888
1.0972	23	11.657	1.2669	58	29.395
1.1017	24	12.164	1.2721	59	29.902
1.1062	25	12.670	1.2773	60	30.408
1.1107	26	13.177	1.2825	61	30.915
1.1153	27	13.684	1.2877	62	31.422
1.1199	28	14.191	1.2929	63	31.929
1.1246	29	14.698	1.2981	64	32.436
1.1292	30	15.204	1.3034	65	32.942
1.1339	31	15.711	1.3087	66	33.449
1.1386	32	16.218	1.3140	67	33.956
1.1433	33	16.725	1.3193	68	34.463
1.1480	34	17.232	1.3246	69	34.970
1.1527	35	17.738	1.3300	70	35.476

Specific Gravity of Solutions of Sodium Chloride at 15° C.

Specific gravity.	Per cent. NaCl.	Specific gravity.	Per cent. NaCl.	Specific gravity.	Per cent. NaCl.
1.00725	1.1	1.07335	10.0	1.14315	19.0
1.01450	1.2	1.08097	11.0	1.15107	20.0
1.02174	1.3	1.08859	12.0	1.15931	21.0
1.02899	1.4	1.09622	13.0	1.16755	22.0
1.03624	1.5	1.10384	14.0	1.17580	23.0
1.04366	1.6	1.11146	15.0	1.18404	24.0
1.05108	1.7	1.11938	16.0	1.19228	25.0
1.05851	1.8	1.12730	17.0	1.20098	26.0
1.06593	1.9	1.13523	18.0	1.20433	26.395

SPECIFIC GRAVITY OF GASES AND VAPORS.

Gas or vapor.	Formula.	Molecular weight.	Specific gravity. (air=1).	Weight of one liter in grams at at 0° C. and 760 mm.
Acetone	C_2H_6O	58.0	2.0025	2.5896
Acetylene	C_2H_2	26.0	0.9200	1.1650
Air	....	...	1.0000	1.29387
Aldehyde	C_2H_4O	44.0	1.5320	1.9811
Ammonia	NH_3	17.0	0.5960	0.7707
Amylic alcohol	$C_5H_{12}O$	88.0	3.1470	4.0696
Arsenic	As_4	300.0	10.3900	13.4362
Arsenious anhydride	As_2O_3	198.0	3.8500	7.9105
Arsine	AsH_3	78.0	2.6950	3.4851
Benzene	C_6H_6	78.0	2.7700	3.5821
Bromine	Br_2	160.0	5.3933	6.8697
Butane	C_4H_{10}	58.0	2.0041	2.5914
Carbon bisulphide	CS_2	76.0	2.6450	3.4204
Carbon dioxide	CO_2	44.0	1.5290	1.9662
Carbon monoxide	CO	28.0	0.9674	1.2510
Carbon oxychloride	$COCl_2$	99.0	3.4163	4.4174
Carbon oxysulphide	COS	60.0	2.0748	2.6828
Chlorine	Cl_2	71.0	2.448	3.1801
Chlorine cyanide	$CNCl$	61.5	2.1244	2.7473
Chloroform	$CHCl_3$	119.5	4.2150	4.4507
Cyanogen	$(CN)_2$	52.0	1.8064	2.3360
Ethane	C_2H_6	30.0	1.0366	1.3404
Ether	$C_4H_{10}O$	74.0	2.5650	3.3170
Ether acetic	$C_4H_8O_2$	88.0	3.0670	3.9662
Ethylic alcohol	C_2H_6O	46.0	1.6133	2.0862
Ethylene	C_2H_4	28.0	0.9674	1.2510
Hydrobromic acid	HBr	81.0	2.7310	3.5316
Hydrochloric acid	HCl	36.5	1.2474	1.6131
Hydrocyanic acid	HCN	27.0	0.9456	1.2228
Hydrofluoric acid	HF	20.0	0.6930	0.8960
Hydrogen	H_2	2.0	0.06926	0.08958
Hydrogen sulphide (sulphuretted hydrogen)	H_2S	34.0	1.1921	1.5416
Hydroiodic acid	HI	128.0	4.4330	5.7456
Iodine	I_2	254.0	8.7160	11.2710
Mercury	Hg	200.0	6.9760	9.0210
Methane	CH_4	16.0	0.5560	0.7155
Methylic alcohol	CH_4O	32.0	1.1200	1.4483
Nitric oxide	NO	30.0	1.0390	1.3436
Nitrogen	N_2	28.0	0.97137	1.25617
Nitrous oxide	N_2O	44.0	1.5269	1.9745
Oxygen	O_2	32.0	1.1056	1.4298
Phosphine (phosphuretted hydrogen)	PH_3	34.0	1.1850	1.5350
Phosphorus	P_4	124.0	4.3550	5.6318
Phosphorus pentachloride	PCl_5	208.5	3.6500	4.7201
Phosphorus trichloride	PCl_3	137.5	4.7420	6.1299
Propane	C_3H_8	44.0	1.5204	1.9660
Selenium	Se_2	158.0	5.7000	7.0229
Selenium hydride	SeH_2	81.0	2.7846	3.6011

SPECIFIC GRAVITY OF GASES AND VAPORS—*Continued.*

Gas or vapor.	Formula.	Molecular weight.	Specific gravity: (air=1).	Weight of one liter in grams at at 0° C. and 760 mm.
Silicon chloride	$SiCl_4$	169.5	5.9390	7.6208
Silicon fluoride	SiF_4	104.0	3.6000	4.6554
Steam	H_2O	18.0	0.6235	0.8063
Sulphur	Si	64.0	2.2000	2.8430
Sulphuric acid	H_2SO_4	98.0	2.1500	2.7803
Sulphuric acid, anhydrous	SO_3	80.0	2.7630	3.5730
Sulphurous acid, anhydrous	SO_2	64.0	2.234	2.8689
Tellurium	Te_2	256.0	8.9160	11.5310
Tellurium hydride	TeH_2	130.0	4.5276	5.8550

COMPARISON OF THE DEGREES OF BAUMÈ'S HYDROMETER WITH THE REAL SPECIFIC GRAVITIES.

1. For Liquids Heavier than Water.[1]

Degrees.	Specific gravity.	Degrees.	Specific gravity.	Degrees.	Specific gravity.
0	1.000	26	1.206	52	1.520
1	1.007	27	1.216	53	1.535
2	1.013	28	1.226	54	1.551
3	1.020	29	1.236	55	1.567
4	1.027	30	1.246	56	1.583
5	1.034	31	1.256	57	1.600
6	1.041	32	1.267	58	1.617
7	1.048	33	1.277	59	1.634
8	1.056	34	1.288	60	1.652
9	1.063	35	1.299	61	1.670
10	1.070	36	1 310	62	1.689
11	1.078	37	1.322	63	1.708
12	1.085	38	1.333	64	1.727
13	1.094	39	1.345	65	1.747
14	1.101	40	1.357	66	1.767
15	1.109	41	1.369	67	1.788
16	1.118	42	1.382	68	1.809
17	1.126	43	1.395	69	1.831
18	1.134	44	1.407	70	1.854
19	1.143	45	1.420	71	1.877
20	1.152	46	1.434	72	1.900
21	1.160	47	1.448	73	1.924
22	1.169	48	1.462	74	1.949
23	1.178	49	1.476	75	1.974
24	1.188	50	1.490	76	2.000
25	1.197	51	1.504		

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[1] The Table of Comparison of the Degrees of Baumè's Hydrometer with the real Specific Gravities for liquids lighter than water will be found on page 371.

OF THE PROPORTION BY WEIGHT OF ABSOLUTE OR REAL ALCOHOL IN 100 PARTS OF SPIRITS OF DIFFERENT SPECIFIC GRAVITIES.

(MENDELEJEFF.)[1]

Specific gravity at 15° C.	Per cent. of real alcohol.	Specific gravity. at 15° C.	Per cent. of real alcohol.	Specific gravity at 15° C.	Per cent. of real alcohol.
0.9991	0.5	0.9501	34	0.8773	68
0.9981	1	0.9491	35	0.8750	69
0.9963	2	0.9473	36	0.8726	70
0.9945	3	0.9455	37	0.8702	71
0.9928	4	0.9436	38	0.8678	72
0.9912	5	0.9417	39	0.8655	73
0.9896	6	0.9397	40	0.8631	74
0.9881	7	0.9377	41	0.8607	75
0.9867	8	0.9357	42	0.8582	76
0.9853	9	0.9336	43	0.8558	77
0.9839	10	0.9316	44	0.8534	78
0.9826	11	0.9294	45	0.8510	79
0.9813	12	0.9273	46	0.8485	80
0.9801	13	0.9251	47	0.8460	81
0.9789	14	0.9230	48	0.8435	82
0.9777	15	0.9208	49	0.8410	83
0.9765	16	0.9186	50	0.8386	84
0.9753	17	0.9164	51	0.8360	85
0.9741	18	0.9142	52	0.8335	86
0.9728	19	0.9119	53	0.8309	87
0.9716	20	0.9097	54	0.8283	88
0.9704	21	0.9074	55	0.8257	89
0.9691	22	0.9052	56	0.8230	90
0.9678	23	0.9029	57	0.8203	91
0.9665	24	0.9097	58	0.8176	92
0.9651	25	0.8983	59	0.8149	93
0.9637	26	0.8960	60	0.8120	94
0.9623	27	0.8937	61	0.8092	95
0.9608	28	0.8914	62	0.8063	96
0.9593	29	0.8890	63	0.8034	97
0.9577	30	0.8867	64	0.8004	98
0.9561	31	0.8844	65	0.7973	99
0.9544	32	0.8820	66	0.7942	100
0.9527	33	0.8797	67		

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[1] Pogg. Annallen, 138, p. 103.

OF THE PROPORTION BY VOLUME OF ABSOLUTE OR REAL ALCOHOL IN 100 VOLUMES OF SPIRITS OF DIFFERENT SPECIFIC GRAVITIES AT 15.5° C. (MENDELEJEFF.)[1]

100 volumes spirits.		100 volumes spirits.		100 volumes spirits.	
Specific gravity.	Contain volumes of real alcohol.	Specific gravity.	Contain volumes of real alcohol.	Specific gravity.	Contain volumes of real alcohol.
1.0000	0	0.9604	34	0.8950	68
0.9985	1	0.9591	35	0.8925	69
0.9970	2	0.9577	36	0.8901	70
0.9956	3	0.9563	37	0 8876	71
0.9942	4	0.9548	38	0.8851	72
0.9928	5	0.9534	39	0.8826	73
0.9915	6	0.9518	40	0.8800	74
0.9902	7	0.9503	41	0.8774	75
0.9889	8	0.9486	42	0.8747	76
0.9877	9	0.9470	43	0.8721	77
0.9866	10	0.9454	44	0.8694	78
0.9854	11	0.9436	45	0.8667	79
0.9844	12	0.9419	46	0.8640	80
0.9832	13	0.9400	47	0.8611	81
0.9822	14	0.9382	48	0.8583	82
0.9811	15	0.9364	49	0.8554	83
0.9801	16	0.9344	50	0.8525	84
0.9790	17	0.9325	51	0.8496	85
0.9781	18	0.9305	52	0.8466	86
0.9771	19	0.9285	53	0.8435	87
0.9761	20	0.9265	54	0.8404	88
0.9751	21	0.9244	55	0.8372	89
0.9741	22	0.9222	56	0.8340	90
0.9731	23	0.9201	57	0.8306	91
0.9720	24	0.9180	58	0.8272	92
0.9709	25	0.9158	59	0.8236	93
0.9699	26	0.9139	60	0.8199	94
0.9688	27	0.9113	61	0.8161	95
0.9677	28	0.9090	62	0.8121	96
0.9667	29	0.9067	63	0.8080	97
0.9654	30	0.9045	64	0.8035	98
0.9642	31	0.9022	65	0.7989	99
0.9630	32	0.8997	66	0.7939	100
0.9617	33	0.8974	67		

[1] Pogg. Annallen, 138, 230.

TABLE SHOWING PERCENTAGES OF REAL SULPHURIC ACID (H_2SO_4) CORRESPONDING TO VARIOUS SPECIFIC GRAVITIES OF AQUEOUS SULPHURIC ACID.

Bineau; Otto. Temperature 15° C.

Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.	Specific gravity.	Per cent.
1.8426	100	1.675	75	1.398	50	1.182	25
1.842	99	1.663	74	1.3886	49	1.174	24
1.8406	98	1.651	73	1.379	48	1.167	23
1.840	97	1.639	72	1.370	47	1.159	22
1.8384	96	1.627	71	1.361	46	1.1516	21
1.8376	95	1.615	70	1.351	45	1.144	20
1.8356	94	1.604	69	1.342	44	1.136	19
1.834	93	1.592	68	1.333	43	1.129	18
1.831	92	1.580	67	1.324	42	1.121	17
1.827	91	1.568	66	1.315	41	1.1136	16
1.822	90	1.557	65	1.306	40	1.106	15
1.816	89	1.545	64	1.2976	39	1.098	14
1.809	88	1.534	63	1.289	38	1.091	13
1.802	87	1.523	62	1.281	37	1.083	12
1.794	86	1.512	61	1.272	36	1.0756	11
1.786	85	1.501	60	1.264	35	1.068	10
1.777	84	1.490	59	1.256	34	1.061	9
1.767	83	1.480	58	1.2476	33	1.0536	8
1.756	82	1.469	57	1.239	32	1.0464	7
1.745	81	1.4586	56	1.231	31	1.039	6
1.734	80	1.448	55	1.223	30	1.032	5
1.722	79	1.438	54	1.215	29	1.0256	4
1.710	78	1.428	53	1.2066	28	1.019	3
1.698	77	1.418	52	1.198	27	1.013	2
1.686	76	1.408	51	1.190	26	1.0064	1

TABLE GIVING THE PERCENTAGES OF HYDROCHLORIC ACID CONTAINED IN AQUEOUS SOLUTIONS OF THE GAS OF VARIOUS SPECIFIC GRAVITIES.

Ure. Temperature 15° C.

Specific gravity.	Per cent. HCl.	Specific gravity.	Per cent. HCl.	Specific gravity.	Per cent. HCl.	Specific gravity.	Per cent. HCl.
1.200	40.777	1.1515	30.582	1.1000	20.388	1.0497	10.194
1.1982	40.369	1.1494	30.174	1.0980	19.980	1.0477	9.786
1.1964	39.961	1.1473	29.767	1.0960	19.572	1.0457	9.379
1.1946	39.554	1.1452	29.359	1.1939	19.165	1.0437	8.971
1.1928	39.146	1.1431	28.951	1.0919	18.757	1.0417	8.563
1.1910	38.738	1.1410	28.544	1.0899	18.349	1.0397	8.155
1.1893	38.330	1.1389	28.136	1.0879	17.941	1.0377	7.747
1.1875	37.923	1.1369	27.728	1.0859	17.534	1.0357	7.340
1.1857	37.516	1.1349	27.321	1.0838	17.126	1.0337	6.932
1.1846	37.108	1.1328	26.913	1.0818	16.718	1.0318	6.524
1.1822	36.700	1.1308	26.505	1.0798	16.310	1.0298	6.116
1.1802	36.292	1.1287	26.098	1.0778	15.902	1.0279	5.709
1.1782	35.884	1.1267	25.690	1.0758	15.494	1.0259	5.301
1.1762	35.476	1.1247	25.282	1.0738	15.087	1.0239	4.893
1.1741	35.068	1.1226	24.874	1.0718	14.679	1.0220	4.486
1.1721	34.660	1.1206	24.466	1.0697	14.271	1.0200	4.078
1.1701	34.252	1.1185	24.058	1.0677	13.863	1.0180	3.670
1.1681	33.845	1.1164	23.650	1.0657	13.456	1.0160	3.262
1.1661	33.437	1.1143	23.242	1.0637	13.049	1.0140	2.854
1.1641	33.029	1.1123	22.834	1.0617	12.641	1.0120	2.447
1.1620	32.621	1.1102	22.426	1.0597	12.233	1.0100	2.039
1.1599	32.213	1.1082	22.019	1.0577	11.825	1.0080	1.631
1.1578	31.805	1.1061	21.611	1.0557	11.418	1.0060	1.124
1.1557	31.398	1.1041	21.203	1.0537	11.010	1.0040	0.816
1.1536	30.990	1.1020	20.796	1.0517	10.602	1.0020	0.408

Percentages and Gravity of Nitric Acid.

TABLE SHOWING THE PERCENTAGES OF NITRIC ACID (HNO_3) IN AQUEOUS SOLUTIONS OF VARIOUS SPECIFIC GRAVITIES.

Kolb, Ann. Chem. Phys., 4, 136. Temperature 15° C.

Per cent. HNO_3.	Specific gravity.	Per cent. HNO_3.	Specific gravity.	Per cent. HNO_3.	Specific gravity.	Per cent. HNO_3.	Specific gravity.
100.00	1.530	80.96	1.463	59.59	1.372	39.00	1.244
99.84	1.530	80.00	1.460	58.88	1.368	37.95	1.237
99.72	1.530	79.00	1.456	58.00	1.363	36.00	1.225
99.52	1.529	77.66	1.451	57.00	1.358	35.00	1.218
97.89	1.523	76.00	1.445	56.10	1.353	33.86	1.211
97.00	1.520	75.00	1.442	55.00	1.346	32.00	1.198
96.00	1.516	74.01	1.438	54.00	1.341	31.00	1.192
95.27	1.514	73.00	1.435	53.81	1.339	30.00	1.185
94.00	1.509	72.39	1.432	53.00	1.335	29.00	1.179
93.01	1.506	71.24	1.429	52.33	1.331	28.00	1.172
92.00	1.503	69.96	1.423	50.99	1.323	27.00	1.166
91.00	1.499	69.20	1.419	49.97	1.317	25.71	1.157
90.00	1.495	68.00	1.414	49.00	1.312	23.00	1.138
89.56	1.494	67.00	1.410	48.00	1.304	20.00	1.120
88.00	1.488	66.00	1.405	47.18	1.298	17.47	1.105
87.45	1.486	65.07	1.400	46.64	1.295	15.00	1.089
86.17	1.482	64.00	1.395	45.00	1.284	13.00	1.077
85.00	1.478	63.59	1.393	43.53	1.274	11.41	1.067
84.00	1.474	62.00	1.386	42.00	1.264	7.22	1.045
83.00	1.470	61.21	1.381	41.00	1.257	4.00	1.022
82.00	1.467	60.00	1.374	40.00	1.251	2.00	1.010

NORMAL SOLUTIONS.

Normal sulphuric acid contains 49.0 grams H_2SO_4 per liter. One cc. contains 0.049 gram H_2SO_4.

Normal hydrochloric acid contains 36.37 grams HCl per liter. One cc. contains 0.036 gram HCl.

Normal nitric acid contains 63.0 grams HNO_3 per liter. One cc. contains 0.063 gram HNO_3.

Normal oxalic acid contains 63.0 grams $C_2O_4H_2.2H_2O$ per liter. One cc. contains 0.045 gram $C_2O_4H_2$.

Normal potassium hydroxide contains 56.0 grams KOH per liter. One cc. contains 0.056 gram KOH.

Normal sodium hydroxide contains 40.0 grams NaOH per liter. One cc. contains 0.040 gram NaOH.

Normal sodium carbonate contains 53.0 grams Na_2CO_3 per liter. One cc. contains 0.053 gram Na_2CO_3.

One-half normal ammonium hydroxide contains 8.5 grams NH_3 per liter. One cc. contains 0.0085 gram NH_3.

One-tenth normal potassium permanganate contains 3.156 grams $K_2Mn_2O_8$ per liter. One cc. contains 0.0008 gram oxygen.

One-tenth normal potassium bichromate contains 4.913 grams $K_2Cr_2O_7$ per liter. One cc. contains 0.0049 gram $K_2Cr_2O_7$.

One-tenth normal iodine contains 12.65 grams I per liter. One cc. equivalent to $\begin{cases} \text{0.01265 gram iodine.} \\ \text{0.02480 gram } Na_2S_2O_3.5H_2O. \end{cases}$

One-tenth normal sodium thiosulphate contains 24.8 grams $Na_2S_2O_3.5H_2O$ per liter. One cc. equivalent to $\begin{cases} \text{0.01265 gram iodine.} \\ \text{0.02480 gram } Na_2S_2O_3.5H_2O. \end{cases}$

One-tenth normal silver nitrate contains 16.966 grams $AgNO_3$ per liter. One cc. equivalent to $\begin{cases} \text{0.01076 gram Ag.} \\ \text{0.00354 gram Cl.} \end{cases}$

One-tenth normal sodium chloride contains 5.837 grams NaCl per liter. One cc. equivalent to $\begin{cases} \text{0.0058 gram NaCl.} \\ \text{0.0035 gram Cl.} \end{cases}$

For ammonium molybdate solution consult page 177.

For a magnesia mixture consult page 178.

INDICATORS.

Phenolphthalein—Alcoholic solution 1 : 30. Colorless by acids; red violet by alkalies; also by CO_2.

Methyl orange—Water solution 1 : 1000. Yellow color by alkalies; purple red by mineral acids; not affected by CO_2.

Litmus—Water solution. Blue by alkalies; red by acids.

Cochinelle—Three parts cochinelle; 400 parts H_2O; 100 parts alcohol. Violet by alkalies; yellowish red by acids.

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