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# GAS AND FUEL ANALYSIS FOR ENGINEERS.

A COMPEND FOR THOSE INTERESTED IN THE  
ECONOMICAL APPLICATION OF FUEL.

*PREPARED ESPECIALLY FOR THE USE OF STUDENTS  
at the  
MASSACHUSETTS INSTITUTE OF TECHNOLOGY.*

BY

AUGUSTUS H. GILL, S.B., PH.D.,  
*Professor of Technical Analysis at the  
Massachusetts Institute of Technology, Boston, Mass.  
Author of "A Short Handbook of Oil Analysis,"  
"Engine Room Chemistry."*



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## PREFACE.

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THIS little book is an attempt to present in a concise yet clear form the methods of gas and fuel analysis involved in testing the efficiency of a boiler plant. Its substance was given originally, in the form of lectures and heliographed notes, to the students in the courses of Chemical, Mechanical, and Electrical Engineering, but in response to requests it has been deemed expedient to give it a wider circulation.

At the time of its conception, nothing of the kind was known to exist in the English language; in German we now have the excellent little book of Dr. Ferdinand Fischer, "Taschenbuch für Feuerungs-Techniker."

The present book is the result of six years' experience in the instruction of classes of about one hundred students. It is in no sense a copy of any other work, nor is it a mere compilation. The author has in every case endeavored to give credit where anything has been taken from outside sources; it is, how-

ever, difficult to credit single ideas, and if he has been remiss in this respect it has been unintentional.

The study of flue-gas analysis enables the engineer to investigate the various sources of loss; and if this compend stimulates and renders easy such investigation, the writer's purpose will have been accomplished. The necessary apparatus can be obtained from the leading dealers in New York City.

The author wishes to acknowledge his indebtedness to our former Professor of Analytical Chemistry, Dr. Thomas M. Drown, and to Mrs. Ellen H. Richards, by whose efforts the department of Gas Analysis was established.

He will also be grateful for any suggestions or corrections from the profession.

MASSACHUSETTS INSTITUTE OF TECHNOLOGY,  
BOSTON, November, 1896.

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## PREFACE TO THE SIXTH EDITION.

THE changes made in the present edition consist in the addition of Specifications for the Purchase of Coal and Fuel Oil and of the Sampling of Fuel Oil and Natural Gas.

As in the past, minor additions and corrections have been made where necessary to bring the book up to present practice.

MASSACHUSETTS INSTITUTE OF TECHNOLOGY,  
Boston, November, 1911.

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# GAS AND FUEL ANALYSIS.

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## CHAPTER I.

### INTRODUCTION AND METHODS OF SAMPLING.

UNTIL within recent years, the mechanical engineer in testing a boiler plant has been compelled to content himself with the bare statement of its efficiency, little or no idea being obtained as to the apportionment of the losses. Knowing the composition and temperature of the chimney-gases and the analysis of the coal and ash, the loss due to the formation of carbonic oxide, to the imperfect combustion of the coal, to the high temperature of the escaping gases, can each be determined and thus a basis for their reduction to a minimum established.

By the simple analysis of the chimney-gases and determination of their temperature, a very good idea of the efficiency of the plant can be obtained previous to making the engineering test. For example, in a test which the author made in connection with his students, the efficiency was increased from 58 to 70 per cent, upon the results of the gas analysis alone.

To this end a representative sample must be collected according to the method about to be described.

### SAMPLING.

Before proceeding to take a sample of the gas, the plant—for example, a boiler setting—from which the gas is to be taken should be thoroughly inspected, and all apertures by which the air can enter, carefully stopped up. A suitable tube is then inserted air-tight in the gas-duct, connected with the sampling or gas apparatus, and suction applied, thus drawing the gas out. Cork, putty, plaster of Paris, wet cotton-waste, or asbestos may be used to render the joint gas-tight. The place of insertion should be chosen where the gas will be most completely mixed and least contaminated with air. The oil-bath containing the thermometer is similarly inserted near the gas-tube, and the temperature read from time to time.

**1. Tubes.**—The tubes usually employed are Bohemian-glass combustion tubing or water-cooled metal tubes; those of porcelain or platinum are also sometimes used. Glass and porcelain tubes when subjected to high temperatures must be previously warmed or gradually inserted: the former may be used up to temperatures of  $600^{\circ}$  C. ( $1200^{\circ}$  F.). Uncooled metal tubes, other than those of platinum, should under no circumstances be used.\*

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\* Fischer, "Technologie der Brennstoffe," 1880, p. 221, states that the composition of a gaseous mixture was changed from 1.5 to 26.0 per cent carbon dioxide, by the passage through an iron tube heated to a dull red heat, the carbonic oxide originally present reducing the iron oxide with the formation of carbon dioxide.



The metal tube with the water cooling is made as shown in Fig. 1, *c* being a piece of brass pipe 3 feet long,  $1\frac{1}{4}$  inches outside diameter, *b* the same length,  $\frac{7}{8}$  inch in diameter, and *a*  $\frac{1}{4}$  inch in diameter. The water enters at *d* and leaves at *e*. The walls of

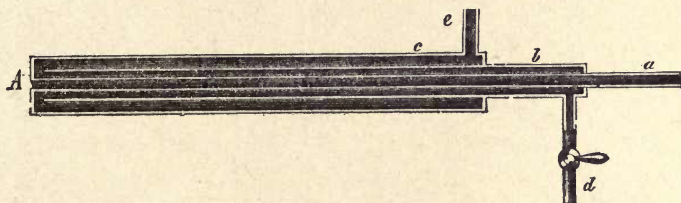


FIG. 1.—GAS-SAMPLING TUBE.

the tubes are  $\frac{1}{16}$  inch thick. The joint at *A* should be brazed; the others may be soldered.

Platinum tubes from their high cost and small bore are seldom used; they are attacked by carbon, cyanogen, arsenic, and metallic vapors.

**2. Apparatus for the Collection of Samples.**—A convenient sampling apparatus is shown in Fig. 2. It may be made from a liter separatory funnel—instead of the bulb there shown—fitted with a rubber stopper carrying a tube passing to the bottom and a T tube; both of these, except where sulphur-containing gases are present, can advantageously be made of  $\frac{3}{16}$ -inch lead pipe. The stopper should not be fastened down with wire between the tubes after the manner of wiring effervescent drinks, as this draws the rubber away from the tubes and occasions a leak. The fastening shown consists of a brass plate fitting upon the top of the stopper, provided with screws and nuts which pass through a wire around

the neck of the separatory. A chain fastened to the plate serves as a convenient method of handling it.

In using the apparatus, the bulb is filled with water by connecting the stem with the water-supply and opening one of the pinchcocks upon the T tube; the

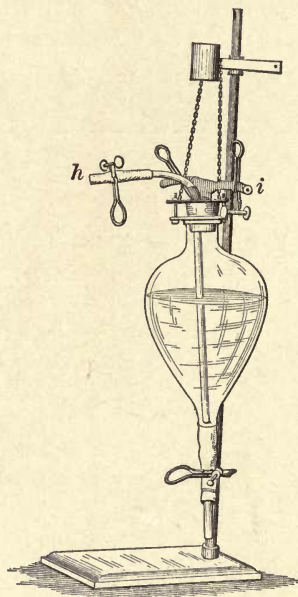


FIG. 2.—SAMPLING APPARATUS.

water thus entering from the bottom forces the air out before it. One branch of the T is connected with the sampling-tube and the other with the suction-pump, the stopcocks being open, and a current of gas drawn down into the pump; upon opening the cock upon the stem, the water runs out, drawing a small portion of the gas-current passing through the T after it into the bulb. It is then taken to a convenient

place for analysis, the tube *h* connected with a head of water, a branch of the T *i*, with the gas apparatus, and a sample of gas forced over the letter for analysis.

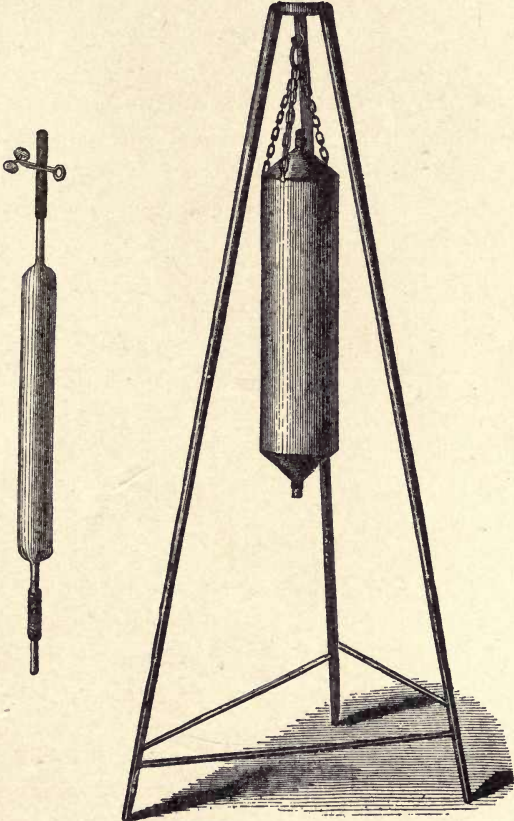


FIG. 4.—GAS-TUBE. FIG. 3.—SAMPLING APPARATUS FOR MINE-GASES.

Enough water should be left in the bulb to seal the stopcock on the bottom and prevent leakage. This apparatus is better adapted for the needs of the class-

room than for actual practice, as it enables the same sample to be given to eight or ten students. As has been shown by several years' experience, the water exercises no appreciable solvent action upon the gaseous mixture in the time—about half an hour—necessary to collect and distribute the samples. It is often necessary to attach about a yard of  $\frac{1}{4}$ -inch rubber tubing to the stem of the bulb to prevent air being sucked up through it when taking a sample.

In the actual boiler-test it is preferable to insert a T instead of this apparatus in the gas-stream, connect the gas apparatus to the free branch of this T, and draw the sample. In making connections with gas apparatus the air in the rubber connectors should be displaced with water by means of a medicine-dropper.

In the Saxon coal-mines, zinc cans of ten liters capacity, of the form shown in Fig. 3, are used by Winkler for sampling the mine-gases; they are carried down filled with water and this allowed to run out, and the gas thus obtained brought into the laboratory and analyzed. Small samples of gas may very well be taken in tubes of 100 cc. capacity like Fig. 4, the ends of which are closed with rubber connectors and glass plugs. Rubber bags are not to be recommended for the collection and storage of gas for analysis, as they permit of the diffusion of gases, notably hydrogen.

**3. Apparatus for Producing Suction.**—I. WATER-PUMPS—(a) Jet-pumps, depending for their action upon a considerable head of water, and (b) those depending rather upon a sufficient fall of water.



(a) *Jet-pumps*.—The Richards' jet pump\* is shown in section in Fig. 5 and much resembles a boiler injector; it consists of a water-jet *w*, a constriction or waist *a*, a waste-tube *o*, and a tube for the inspiration of air. The jet of water forms successive pistons across *a*, drawing the air in with it and is broken up into foam by the zigzag tube *o*.

This pump is known in Germany as Muencke's, and in England as Wing's; Chapman's pump is also a modified form.

It may be easily constructed in glass, the jets passing through rubber stoppers which are wired down, thus admitting of adjustment to the conditions under which it has to work.†

(b) *Fall-pumps*.—Bunsen's pump, Fig. 6, consists of a wide glass tube *A*, drawn out at the bottom for connection with a  $\frac{1}{4}$ -inch lead pipe *b*, and at the top for connection with *c*, the tube through which the air is drawn; this tube is usually fused in, although it may be connected with rubber; *a* is a rubber tube provided with screw cocks connected with the water-supply; *d* is connected with a mercury column, and the vessel *B* serves for the retention of any water which might be drawn back into the apparatus evacuated.

The tube *b* for the best results should be 32 feet in length, equal to the height of a column of water supported by the atmosphere, although for the ordinary purposes of gas-sampling it may be shorter.

When water is admitted through *a* it fills *b*, acting

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\* Richards, Am. Jour. of Science (3), 8, 412; Trans. Am. Inst. Min. Engrs., 6, 492 (1874).

† The pump will also work well using steam.

as a continually falling piston drawing the current of air through *e* and its connections. These various forms of water-pumps should give a vacuum repre-

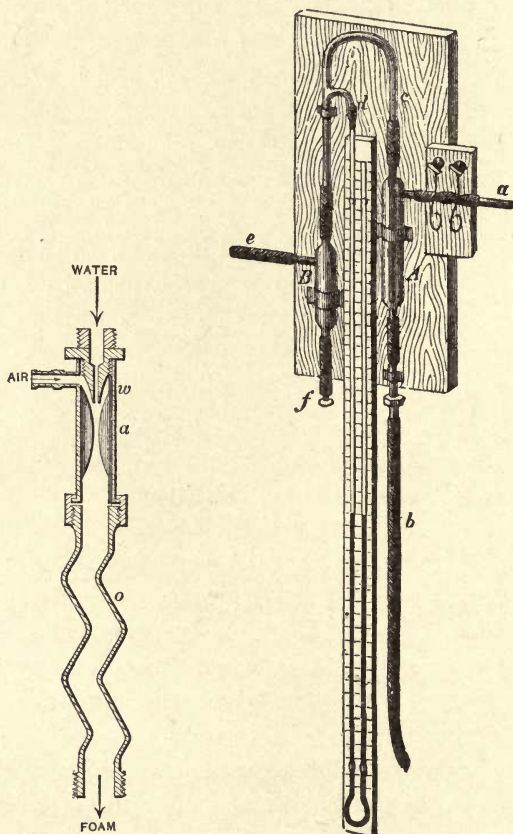


FIG. 5.—RICHARDS' JET-PUMP. FIG. 6.—BUNSEN'S PUMP.

sented by the height of the barometer less the tension of aqueous vapor at the temperature at which they are used, or about 29 inches of mercury.

II. STEAM-PUMPS.—Kochinke describes the apparatus in use in the Muldner Hütten in Freiberg, shown at one-fifth size in Fig. 7. It consists of a glass tube drawn down to an opening 6 mm. in diam-

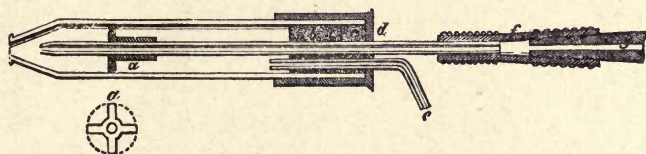


FIG. 7.—STEAM AIR-PUMP.

eter; concentric with this, and held in place by the washer *a*, is the steam-jet 2 mm. in diameter, passing through the cork *b*, the cement *c*, and covering *d*. It is connected with the steam-pipe at *g* by webbed rubber tubing *f*; the air enters at *e*. This is said to give very good results and be economical in use of steam.

In case neither water nor steam be available, recourse must be had to the ordinary rubber syringe-bulbs, provided with suitable valves, obtainable at any rubber store, or to a bottle aspirator. This consists of two one-gallon bottles, provided with doubly perforated rubber stoppers, carrying tubes of glass or lead bent at right angles. In each bottle one of these tubes passes nearly to the bottom, and these are connected together by a piece of rubber tubing a yard long, carrying a screw pinchcock. The other tube in each case stops immediately under the stopper. Upon filling one of the bottles with water, inserting the stopper and blowing strongly through the short tube, water will fill the long tubes thus forming a siphon,

and upon lowering the empty bottle, a current of air will be sucked in through the short tube originally blown into; this may be regulated by the screw pinchcock.

. In inserting the gas-sampling tube care should be taken not to insert it so close to the source of heat as to draw out the gases in a dissociated, i.e. partly decomposed, condition.

In case of very smoky fuels it is well to filter the gases through rolls of fine wire gauze or asbestos; in sucking them through a washing-bottle, the water may change the composition of the sample.



## CHAPTER II.

### APPARATUS FOR THE ANALYSIS OF CHIMNEY-GASES.

IN the writer's opinion the apparatus which is best adapted for this purpose is that of Orsat; it is readily portable, not liable to be broken, easy to manipulate, sufficiently accurate, and—in the modification about to be described—always ready for use, there being no stopcocks to stick fast.

As the Bunte and Elliott apparatus are also used for this purpose, they too will be described.

Fischer's apparatus, using mercury, is rather too difficult for the average engineer; Hempel's or Morehead's\* apparatus for the analysis of illuminating-gas might also be used; it is, however, not customary.

#### ORSAT APPARATUS.

**Description.**—The apparatus Fig. 8, is enclosed in a case to permit of transportation from place to place; furthermore, the measuring-tube is jacketed with water to prevent changes of temperature affecting the gas-volume. The apparatus consists essentially of the levelling-bottle *A*, the burette *B*, the pipettes *P'*, *P''*, *P'''*, and the connecting tube *T*.

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\* No. 143 Lake Street, Chicago.



**Manipulation.**—The reagents in the pipettes should be adjusted in the capillary tubes to a point on the stem about midway between the top of the pipette and the rubber connector. This is effected by opening wide the pinchcock upon the connector, the

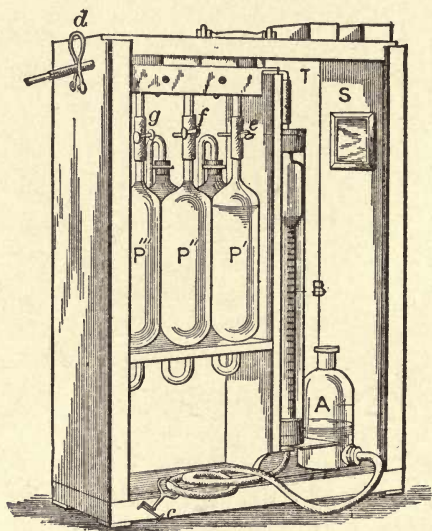


FIG. 8.—ORSAT'S GAS APPARATUS.

bottle being on the table, and very gradually lowering the bottle until the reagent is brought to the point above indicated. Six inches of the tubing used correspond to but 0.1 cc., so that an error of half an inch in adjustment of the reagent is without influence upon the accuracy of the result. The reagents having been thus adjusted, the burette and connecting tube are completely filled with water by opening *d* and raising the levelling-bottle. The apparatus is now

ready to receive a sample of gas (or air for practice). In case a flue-gas is to be analyzed  $d$  is connected with  $i$ , Fig. 2,  $A$  lowered and about 102 cc. of the gas forced over by opening  $h$ ; or  $d$  may be connected with a T joint in the gas-stream; the burette after filling is allowed to drain one minute by the sand-glass,  $c$  snapped upon its rubber tube, and the bottle  $A$  raised to the top of the apparatus. By gradually opening  $c$  the water is allowed to run into the burette until the lower meniscus stands upon the 100 or 0 mark (according to the graduation of the apparatus). The gas taken is thus compressed into the space occupied by 100 cc., and by opening  $d$  the excess escapes. Open  $c$  and *bring the level of the water in the bottle to the same level as the water in the burette* and take the reading, which should be 100 cc. Special attention is called to this method of reading: if the bottle be raised, the gas is compressed; if lowered, it is expanded.

**Determination of Carbon Dioxide.**—The gas to be analyzed is invariably passed first into pipette  $P'$ , containing potassium hydrate for the absorption of carbon dioxide, by opening  $e$  and raising  $A$ . The gas displaces the reagent in the front part of the pipette, laying bare the tubes contained in it, which being covered with the reagent present a large absorptive surface to the gas; the reagent moves into the rear arm of the pipette, displacing the air over it into the flexible rubber bag which prevents its diffusion into the air. The gas is forced in and out of the pipette by raising and lowering  $A$ , the reagent finally brought approximately to its initial point on the stem of the

pipette, the burette allowed to drain one minute, and the reading taken. The difference between this and the initial reading represents the cubic centimeters of carbon dioxide present in the gas. To be certain that all the carbon dioxide is removed, the gas should be passed a second time into  $P'$  and the reading taken as before; these readings should agree within 0.1 per cent.

**Determination of Oxygen.**—The residue from the absorption of carbon dioxide is passed into the second pipette,  $P''$ , containing an alkaline solution of potassium pyrogallate, until no further absorption will take place. The difference between the reading obtained and that after the absorption of carbon dioxide, represents the number of cubic centimeters of oxygen present.

**Determination of Carbonic Oxide.**—The residue from the absorption of oxygen is passed into the third pipette,  $P'''$ , containing cuprous chloride, until no further absorption takes place; that is, in this case until readings agreeing exactly (not merely to 0.1) are obtained. The difference between the reading thus obtained and that after the absorption of oxygen, represents the number of cubic centimeters of carbonic oxide present.

**Determination of Hydrocarbons.**—The residue left after all absorptions have been made may consist, in addition to nitrogen, the principal constituent, of hydrocarbons and hydrogen. Their determination is difficult for the inexperienced, and, if desired, a sample of the flue-gas should be taken, leaving as little water

in the apparatus as possible, and sent to a competent chemist for analysis.

**Accuracy.**—The apparatus gives results accurate to 0.2 of one per cent.

**Time Required.**—About twenty minutes are required for an analysis; two may be made in twenty-five minutes, using two apparatus.

*Notes.*—The method of adjusting the reagents is the only one which has been found satisfactory: if the bottle be placed at a lower level and an attempt made to shut the pinchcock *c* upon the connector at the proper time, it will almost invariably result in failure.

The process of obtaining 100 cc. of gas is exactly analagous to filling a measure heaping full of grain and striking off the excess with a straight-edge; it saves arithmetical work, as cubic centimeters read off represent percent directly.

It often happens when *e* is opened, *c* being closed, that the reagent in *P'* drops, due not to a leak as is usually supposed, but to the weight of the column of the reagent expanding the gas.

The object of the rubber bags is to prevent the access of air to the reagents, those in *P''* and *P'''* absorbing oxygen with great avidity, and hence if freely exposed to the air would soon become useless.

Carbon dioxide is always the first gas to be removed from a gaseous mixture. In the case of air the percentage present is so small, 0.08 to 0.1, as scarcely to be seen with this apparatus. It is important to use the reagents in the order given; if by mistake the gas be passed into the second pipette, it will absorb not only oxygen, for which it is intended, but also carbon



dioxide; similarly if the gas be passed into the third pipette, it will absorb not only carbonic oxide, but also oxygen as well.

The use of pinchcocks and rubber tubes, original with the author, although recommended by Naef,\* is considered by Fischer,† to be inaccurate. The experience of the author, however, does not support this assertion, as they have been found to be fully as accurate as glass stopcocks, and very much less troublesome and expensive.

In case any potassium hydrate or pyrogallate be sucked over into the tube *T* or water in *A*, the analysis is not spoiled, but may be proceeded with by connecting on water at *d*, opening this cock, and allowing the water to wash the tubes out thoroughly. The addition of a little hydrochloric acid to the water in the bottle *A* will neutralize the hydrate or pyrogallate, and the washing may be postponed until convenient.

After each analysis the number of cubic centimeters of oxygen and carbonic oxide should be set down upon the ground-glass slip provided for the purpose. By adding these numbers and subtracting their sum from the absorption capacity (see Reagents) of each reagent, the condition of the apparatus is known at any time, and the reagent can be renewed in season to prevent incorrect analyses.

#### BUNTE APPARATUS.

**Description.**—The apparatus Fig. 9 consists of a burette—bulbed to avoid extreme length—provided

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\* Wagner's Jahresb. 1885, p. 423.

† Technologie d. Brennstoffe, foot note p. 295.



at the top with a funnel *F* and three-way cock *j*, and a cock *l* at the bottom. These stopcocks are best of the Greiner and Friedrichs obliquely bored form. The burette is supported upon a retort-stand with a spring clamp.

A "suction-bottle" *S*, an 8-oz. wide-mouthed bottle, fitted similarly to a wash-bottle, except that the delivery-tube is straight and is fitted with a four-inch piece of  $\frac{1}{4}$ -inch black rubber tubing, serves to withdraw the reagents and water when necessary. A reservoir to contain water at the temperature of the room, fitted with a long rubber tube, should be provided for washing out the reagents and filling the burette.

**Manipulation.** — Before using the apparatus, the keys of the stopcocks should be taken out, wiped dry, together with their seats, and sparingly smeared with vaseline or a mixture of vaseline and tallow and replaced. The completeness of the lubrication can be judged by the transparency of the joint, a thoroughly lubricated joint showing no ground glass. The burette is filled with water by attaching the rubber tube to the tip at *l* and opening the stopcocks at the top and bottom; *j* is connected with the source whence the gas is to be taken, turned to communicate with the burette and opened, about 102 cc. of gas allowed to run in, and *j* and *l* closed.

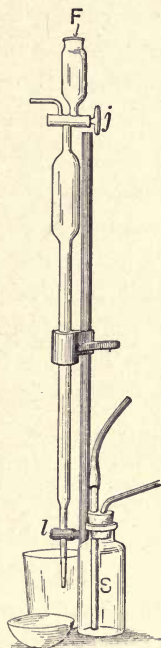


FIG. 9.—BUNTE'S GAS APPARATUS.

The cup *F* is filled with water to the 25-cc. mark, *j* turned to establish communication between it and the burette, the burette allowed to drain one minute by the sand-glass, and the reading taken, the cup being refilled to the mark if necessary. The readings are thus taken under the same pressure each time, i.e., this column of water plus the height of the barometer; and as the latter is practically constant during the analysis, no correction need be applied, it being within the limits of error.

**Determination of Carbon Dioxide.**—The “suction-bottle” is connected with the tip of the burette, *l* opened, and the water carefully sucked out nearly to *l*. The bottle is now disconnected, the burette dismounted from its clamp, using the cup as a handle, and the 25 cc. of water turned out. The tip is immersed under potassium hydrate contained in the No. 3 porcelain dish, and the cock *l* opened, then closed, and the tip wiped clean with a piece of cloth. The burette is now shaken, holding it by the tip and the cup, the thumbs resting upon *j* and *l*; more reagent is introduced, the absorption of the gas causing a diminished pressure, and the operation repeated until no change takes place. The cup is now filled with water, *j* opened, and the reagent completely washed out into an ordinary tumbler placed beneath the burette. Four times filling of *F* should be sufficient for this purpose. The cup is now filled to the 25-cc. mark, *j* opened, and the reading taken as before.

The difference between this reading and the initial represents the number of cubic centimeters of carbon

dioxide; this divided by the volume of the gas taken gives the per cent of this constituent.

**Determination of Oxygen.**—The water is again sucked out, and potassium pyrogallate solution introduced, similarly to potassium hydrate; this is displaced by water, and the reading taken as before. The difference between this and the last reading is the volume of oxygen present.

**Determination of Carbonic Oxide.**—The water is removed for a third time, and acid cuprous chloride solution introduced and the absorption made as before; this is washed out, first with water containing a little hydrochloric acid to dissolve the white cuprous chloride which is precipitated by the addition of water, and finally with pure water, and the reading taken as before. The difference between this and the preceding gives the volume of carbonic oxide present.

*Notes.*—Especial care should be taken not to grasp the burette by the bulb, as this warms the gas and renders the readings inaccurate. The stopcocks can conveniently be kept in the burette by elastic bands of suitable size. When the apparatus is put away for any considerable time, a piece of paper should be inserted between the key and socket of each stopcock to prevent the former from sticking fast. To ascertain when the absorption is complete, the burette is mounted in its clamp and allowed to drain until the meniscus is stationary, the dish containing the reagent raised until the tip is covered, / opened, and any change in level noted. If the meniscus rises, the absorption is incomplete and must be continued; if it remains stationary or falls, the absorption may be regarded as

finished. In case the grease from the stopcocks becomes troublesome inside the burette, it may be removed by dissolving it in chloroform and washing out with alcohol and then with water. The object in sucking the water not quite down to *l*, thus leaving a little water in the burette, is to discover if *l* leaks, the air rushing in causes bubbles.

The object in washing out each reagent and taking all readings over water is to obviate corrections for the tension of aqueous vapor over potassium hydrate, hydrochloric acid, or any of the reagents which might be employed. The tension of aqueous vapor over seven per cent caustic soda is less than over water.

**Accuracy and Time Required.**—The apparatus is rather difficult to manipulate, but fairly rapid—about twenty-five minutes being required for an analysis—and accurate to one tenth of one per cent.

#### ELLIOTT APPARATUS.

**Description.**—The apparatus Fig. 10 consists of a burette holding 100 cc. graduated in tenths of a cubic centimeter and bulbed like the Bunte apparatus—the bulb holding about 30 cc.; it is connected with a levelling-bottle similar to the Orsat apparatus. The top of the burette ends in a capillary stopcock, the stem of which is ground square to admit of close connection with the “laboratory vessel,” an ungraduated tube similar to the burette, except of 125 cc. capacity. The top of this “vessel” is also closed with a capillary stopcock, carrying by a ground-glass joint a thistle-tube *F*, for the introduction of the reagents. The lower end of this “vessel” is closed by a rubber



stopper carrying a three-way cock *o*, and connected with a levelling-bottle *D*. The burette and vessel are held upon a block of wood—supported by a ring stand—by fine copper wire tightened by violin keys.

**Manipulation.**—The ground-glass joints are lubricated as in the Bunte apparatus. The levelling-bottles are filled with water, the stopcocks opened, and the bottles raised until the water flows through the stopcocks *m* and *n*. *m* is connected with the source whence the gas to be analyzed is to be taken, *n* closed, *D* lowered and rather more than 100 cc. drawn in, and *m* closed. *n* is opened, *D* raised and *E* lowered, nearly 100 cc. of gas introduced, and *n* closed; by opening *m* and raising *D* the remainder of the gas is allowed to escape, the tubes being filled with water and *m* closed. *n* is opened and the water brought to the reference-mark; the burette is allowed to drain one minute, the level of the water in *E* is brought to the same level as in the burette, and the reading taken.

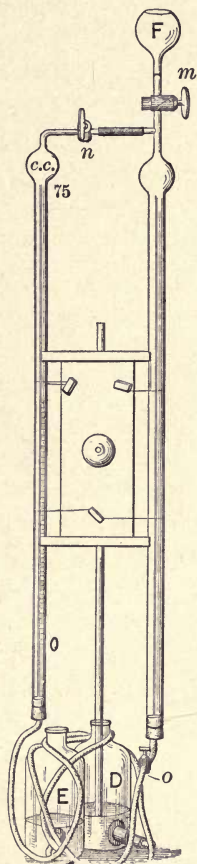


FIG. 10.—ELLIOTT GAS APPARATUS.

**Determination of Carbon Dioxide.**—By raising *E*, opening *n*, and lowering *D*, the gas is passed over into the laboratory vessel; *F* is filled within half an inch



of the top with potassium hydrate, *o* closed, *m* opened, and the reagent allowed to slowly trickle in. A No. 3 evaporating-dish is placed under *o*, and this turned to allow the liquid in the laboratory vessel to run into the dish. At first this is mainly water, and may be thrown away; later it becomes diluted reagent and may be returned to the thistle-tube. When the depth of the reagent in the thistle-tube has lowered to half an inch, it should be refilled either with fresh or the diluted reagent and allowed to run in until the absorption is judged to be complete, and the gas passed back into the burette for measurement. To this end close *o* and then *m*, raise *E*, open *n*, and force some pure water into the laboratory vessel, thus rinsing out the capillary tube. Now raise *D* and lower *E*, shutting *n* when the liquid has arrived at the reference-mark. The burette is allowed to drain a minute, the level of the water in the bottle *E* brought to the same level as the water in the burette, and the reading taken.

**Determination of Oxygen.**—The manipulation is the same as in the preceding determination, potassium pyrogallate being substituted for potassium hydrate; the apparatus requiring no washing out.

**Determination of Carbonic Oxide.**—The laboratory vessel, thistle-tube, and bottle if necessary, are washed free from potassium pyrogallate and the absorption made with acid cuprous chloride similarly to the determination of carbon dioxide. The white precipitate of cuprous chloride may be dissolved by hydrochloric acid.

**Accuracy and Time Required.**—The apparatus is as accurate for absorptions as that of Orsat; it is stated to be much more rapid—a claim which the writer cannot substantiate. It is not as portable, is more fragile, and more troublesome to manipulate, and as the burette is not jacketed it is liable to be affected by changes of temperature.

*Notes.*—In case at any time it is desired to stop the influx of reagent, *o* should be closed first and then *m*; the reason being that the absorption may be so rapid as to suck air in through *o*, *m* being closed.

The stopcock should be so adjusted as to cause the reagent to spread itself as completely as possible over the sides of the burette.

By the addition of an explosion-tube it is used for the analysis of illuminating-gas,\* bromine being used to absorb the "illuminants." Winkler † states that this absorption is incomplete; later work by Treadwell and Stokes, and also Korbuly, ‡ has shown that bromine water, by a purely physical solution, does absorb the "illuminants" completely; Hempel § states that explosions of hydrocarbons made over water are inaccurate, so that the apparatus can be depended upon to give results upon methane and hydrogen only within about two per cent.

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\* Mackintosh, *Am. Chem. Jour.* 9, 294.

† *Zeit. f. Anal. Chem.* 28, 286.

‡ Treadwell's *Quan. Analysis* (Hall's translation), p. 569.

§ *Gasanalytische Methoden*, p. 102.

## GAS-BALANCES.

Under this heading are included various devices either for weighing a volume of the chimney-gas, as the Econometer of Arndt,\* or weighing a globe in an atmosphere of the gas, as the Gas-balance of Custodis.† The Gas-composimeter of Uehling‡ depends upon the laws governing the flow of gases through small apertures.

They are difficult to adjust and keep in adjustment, requiring to be checked frequently by the Orsat apparatus, and are expensive. Their indications are within about half of one per cent of those given by the chemical apparatus. Only the presence of carbon dioxide is indicated by them.

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\* *Zeit. d. Vereins deutsch. Ingenieure*, 37, 801.

† Gill, *Engine Room Chemistry*, pp. 96 and 97.

‡ Poole, *Calorific Power of Fuels*, p. 150, 2d edition (1900).

## CHAPTER III.

### MEASUREMENT OF TEMPERATURE.

IN the majority of cases, the ordinary mercurial thermometer will serve to determine the temperature of the chimney-gases. It should not be inserted naked into the flue, but be protected by a bath of cylinder, or raw linseed oil, contained in a brass or iron tube. These tubes may be half an inch inside diameter and two to three feet in length. Temperatures as high as  $625^{\circ}$  C. have been observed in chimneys; this lasts of course but for a moment, but would be sufficient to burst the unprotected thermometer.

For the observation of higher temperatures, recourse must be had to the "high-temperature thermometers," filled with carbon dioxide under a pressure of about one hundred pounds, giving readings to  $550^{\circ}$  C.\* These may be obtained of the dealers in chemical apparatus; some require no bath, being provided with a mercury-bath carefully contained in a steel tube, and the whole enclosed in a bronze tube.†

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\* Those made by W. Apel, Göttingen, Germany, are about three feet long, the scale occupying about one foot, thus avoiding the necessity of withdrawing the thermometer from the bath for reading. H. J. Green of Brooklyn, N. Y., makes similar ones.

† Hohmann Special Thermometers, made by Hohmann and Maurer Co., 44 High Street, Boston, Mass.



These thermometers should be tested from time to time either by comparison with a standard or by insertion in various baths of a definite temperature. Some of the substances used for these baths are: water, boiling-point  $100^{\circ}$ ; naphthalene, Bpt.  $218^{\circ}$ ; benzophenon, Bpt.  $306^{\circ}$ ; and sulphur,\* Bpt.  $445^{\circ}$ . Care should be taken that the bulb of the thermometer does not dip into the melted substance, but only into the vapor, and that the stem exposure be as nearly as possible that in actual use.

For the measurement of temperatures beyond the range of these thermometers the Le Chatelier thermo-electric pyrometer may be used. This consists of a couple formed by the junction of a platinum and platinum-10% rhodium wire, passing through fire-clay tubes in a porcelain or iron envelope and connected with a galvanometer. The hotter the junction is heated the greater the current and the galvanometer deflection; this latter is determined for several points, naphthalene, sulphur, and copper, Mpt.  $1083^{\circ}$  C., or even platinum,  $1753^{\circ}$  C., and a plot made with galvanometer-readings as abscissæ and temperatures as ordinates. From this the temperature corresponding to any deflection is readily obtained.

The exact description of the instrument and details of calibration are, however, beyond the scope of this work, and the student is referred for these to articles by Le Chatelier, Société Technique de l'Industrie du Gaz, 1890, abstracted in Jour. Soc. Chem. Industry,

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\* In testing the Hohmann thermometers in sulphur-vapor, the bronze tube should be prevented from corrosion by the vapor by a glass envelope.



9, 326, and Holman, Proc. Am. Academy, 1895, p. 234; later works are those of Burgess and Le Chatelier and Boudouard, "High Temperature Measurements" (1912), and also C. L. Norton, "Notes on Heat Measurements" (1902).

An error of  $5^{\circ}$  in the reading of the thermometer affects the final result by about 20 calories.

In case neither of these methods be available nor

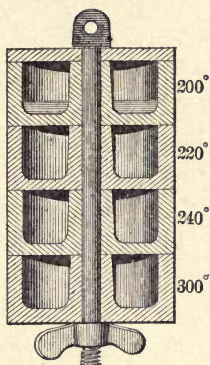


FIG. 11.—MELTING-POINT BOXES.

applicable, use may be made of the melting-points of certain metals or salts contained in small cast-iron boxes, Fig. 11. The melting-points of certain metals and salts are given in Table VII.

## CHAPTER IV.

### CALCULATIONS.

AS has been already stated in the Introduction, the object of analyzing the flue-gases is to ascertain, first, the completeness of the combustion, especially the amount of air which has been used or the "pounds of air per pound of coal," and second, the amount of heat passing up chimney.

**I. To Ascertain the Number of Pounds of Air per Pound of Coal.**—A furnace-gas gives 11.5%  $\text{CO}_2$ , 7.4% O, 0.9% CO, and 80.2% N. Data: atomic weights, O = 16, C = 12; weight liter  $\text{CO}_2$  = 1.966 grs., of O, 1.43 grs., of CO, 1.251 grs. Find the number of grams of each constituent in 100 liters of the furnace-gas, and from this the weight of carbon and weight of oxygen.  $11.5$  (liters  $\text{CO}_2$ )  $\times$  1.97 (wt. liter  $\text{CO}_2$ ) = 22.66 grms.  $\text{CO}_2$ ; now  $\frac{32}{44} \left( \frac{\text{O}_2}{\text{CO}_2} \right)$  of this is oxygen = 16.48 grms., 6.18 grms. is carbon. The weight of free oxygen is  $7.4 \times 1.43 = 10.58$  grms. The weight of carbon and oxygen in the carbonic oxide is  $0.9 \times 1.25 = 1.12$  grms. CO. Now  $\frac{16}{28} \left( \frac{\text{O}}{\text{CO}} \right)$  is oxygen or 0.64 gm., and 0.48 gm. is carbon. There are then present in 100 liters of the gas 27.70 grms. oxygen and 6.66 grms. carbon; corresponding to 120.0 grms. air

to 6.66 grms. carbon, air being 23.1% oxygen by weight; or 18.02  $\left. \begin{array}{l} \text{grms.} \\ \text{lbs.} \end{array} \right\}$  air per  $\left. \begin{array}{l} \text{gm.} \\ \text{lb.} \end{array} \right\}$  carbon. If the coal be 83% carbon, this figure must be diminished accordingly, giving in this case 14.95 lbs. air per lb. of coal. Theory requires 11.54 lbs. air per lb. of carbon, but in practice the best results are obtained by increasing this from 50% to 100%.\*

**2. To Ascertain the Quantity of Heat Passing up Chimney** — Determine the volume of gas generated from one kilo of coal when burned so as to produce the gas the analysis of which has just been made according to the directions given. The chemical analysis of the coal is as follows: moisture 1.5%, sulphur 1.2%, carbon 83%, hydrogen 2.5%, ash 11.4%, oxygen and nitrogen (by difference) 0.4%. Then there are in one kilo of coal 830 grms. carbon, of this suppose but 800 to be burned, the remaining 30 grms. going into the ash; of the 800 grms. 618/666 or 742 grms. produced carbon dioxide, and 48/666 or 58 grms. produced carbonic oxide. From 6.18 grms. carbon were produced 11.5 liters carbon dioxide in the problem in 1; hence 742 grms. would furnish 1381 liters.  $6.18 : 742 :: 11.5 : y$ .  $y = 1381$ . Similarly 58 grms. carbon would furnish 109.0 liters carbonic oxide.  $0.48 : 58 :: 0.90 : z$ .  $z = 109.0$ . The volume of oxygen can be found by the proportion  $11.5 (\% \text{CO}_2) : 7.4 (\% \text{O}) :: 1381 : x$ .  $x = 888$  liters. In the same manner the nitrogen is found to be 9631 liters.  $11.5 : 80.2 :: 1381 : u$ .  $u = 9631$ . One kilo of coal under these conditions furnishes 1.381 cu. meters

\* Scheurer-Kestner, Jour. Soc. Chem. Industry, 7, 616. 0.75 lb. per 1000 B.t.u. Nisbet, Power, 36, 995 (1912).

carbon dioxide, 0.109 c. m. carbonic oxide, 0.888 c. m. oxygen, and 9.631 c. m. nitrogen.

The quantity of heat carried off by each gas is its rise of temperature  $\times$  its weight  $\times$  its specific heat. The specific heats of the various gases are shown in the table below, and for facility in calculation, a column is given obtained by multiplying the weight by the specific heat; multiplying the volumes obtained in the previous calculation by the numbers in this column and by the rise in temperature gives the number of calories (C) that each gas carries away.

TABLE OF SPECIFIC HEATS OF VARIOUS GASES.\*

Name of Gas.	Sp. Heat.	Wt. of Cu. M. Kg.	Sp. Heat $\times$ Wt. of Cu. M.	Log.
Carbon dioxide ( $10^{\circ}$ - $350^{\circ}$ ).	0.234	1.97	0.463	9.6656
“ monoxide.....	0.245	1.26	0.308	9.4886
Oxygen.....	0.217	1.43	0.311	9.4928
Nitrogen.....	0.244	1.26	0.306	9.4857
Aqueous vapor.....	0.480	0.80	0.387	9.5877

In the test the average temperature of the escaping gases was  $275^{\circ}$  C.; that of the air entering the grate was  $25^{\circ}$  C., a rise of temperature of  $250^{\circ}$  C. As shown by the wet-and-dry-bulb thermometer, the air was 50 per cent saturated with moisture.

The calculation of the heat carried away is then for:

	Cu. M.	C.
Carbon dioxide.....	$1.381 \times 250 \times 0.463 =$	160.0
Carbonic oxide.....	$0.109 \times \text{“} \times 0.308 =$	8.4
Oxygen.....	$0.888 \times \text{“} \times 0.311 =$	69.1
Nitrogen.....	$9.631 \times \text{“} \times 0.306 =$	737.0
Total.....	12.009	974.5

\* Fischer, Tech. d. Brennstoffe, p. 267.



There is, however, another gas passing up chimney of which we have taken no cognizance, namely, water-vapor; this comes from the moisture in the coal, from the combustion of hydrogen in the coal, and from the air entering the grate; its volume is calculated as follows:

The moisture in the coal as found by chemical analysis was  $1.5\% = 0.015$  kg.; the hydrogen in the coal was  $2.5\% = 0.025$  kg. The amount of water this forms when burned is nine times its weight,  $0.025$  kg.  $\times 9 = 0.225$  kg. The moisture in the air entering the grate would be, if completely saturated, 22.9 grams per cubic meter, as shown by Table I; it was, however, but 50% saturated. The quantity is then, the volume of air used per kilogram of coal  $\times$  moisture contained in it, or  $12.009 \times 22.9 \times 0.50 = 0.137$  kg. The weight of aqueous vapor passing up chimney per kilogram of coal is  $0.015 + 0.225 + 0.137 = 0.377$  kg.; the quantity of heat that this carries off is  $0.377 \times 250 \times 0.480 = 45.2$  C. The total quantity of heat passing up chimney is then 1019.7 C. The heat of combustion of this coal as found by Mahler's calorimetric bomb was 7220 C.; hence the percentage of heat carried off is  $1020/7220 = 14.1\%$ .

The preceding calculations though correct are tedious, so much so, as to almost preclude their use for an hourly observation of the firing. They should be employed, however, in making the final calculation of a boiler-test, using the averages obtained.

Shields\* has combined the operations in

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\* *Power*, 1908.

**I. Pounds of Air per Pound of Coal** (p. 28), and obtains the following formula:

Pounds of air per pound of coal

$$= 2.31 \frac{\text{Per cent. carbon in coal}}{\text{Per cent. CO}_2 + \text{per cent. CO.}}$$

Similarly, **Per cent. heat lost**

$$= \frac{\text{Per cent. carbon in coal}}{\text{Heating value of coal}} \times \frac{200 + \text{per cent. CO}_2}{\text{Per cent. CO}_2 + \text{per cent. CO}} \times \text{rise in temperature in } ^\circ\text{C.} \times 0.2864.$$

The values found by this equation are 0.5 per cent. low, as no cognizance has been taken of the water vapor.

In rapid work the following formula will be found more applicable: Let *o* and *n* represent the percentages of oxygen and nitrogen found in the chimney-gas; then the ratio of the air actually used to that theoretically necessary is expressed by the formula

$$\frac{21}{21 - \left(\frac{79o}{n}\right)}$$

Applying it in the case of the flue-gas given, it becomes

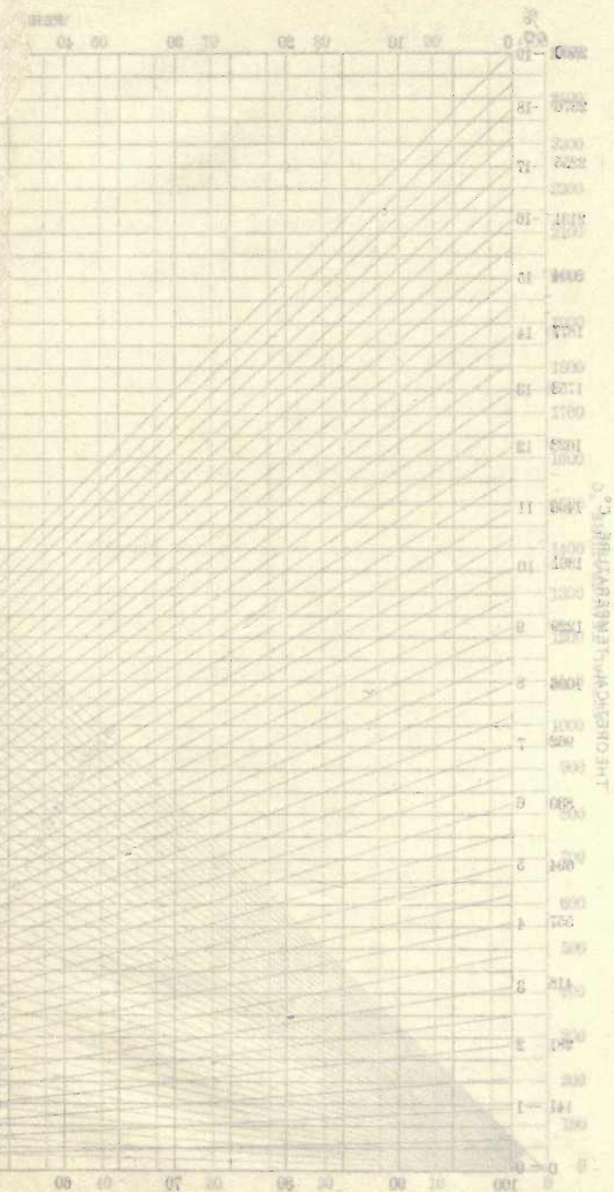
$$\frac{21}{21 - \left(\frac{79 \times 7.4}{80.2}\right)} = \frac{21}{13.7} = 1.533 \text{ ratio.}$$

Multiplying this by 11.54, the theoretical number of pounds of air per pound of carbon, we obtain 17.69 as against 18.02 on page 28.

Bunte\* has given a shorter method for the deter-

\* Jour. f. Gasbeleuchtung, 43, 637 (1900); Abstr. Jour. Soc. Chem. Industry, 19, 887.

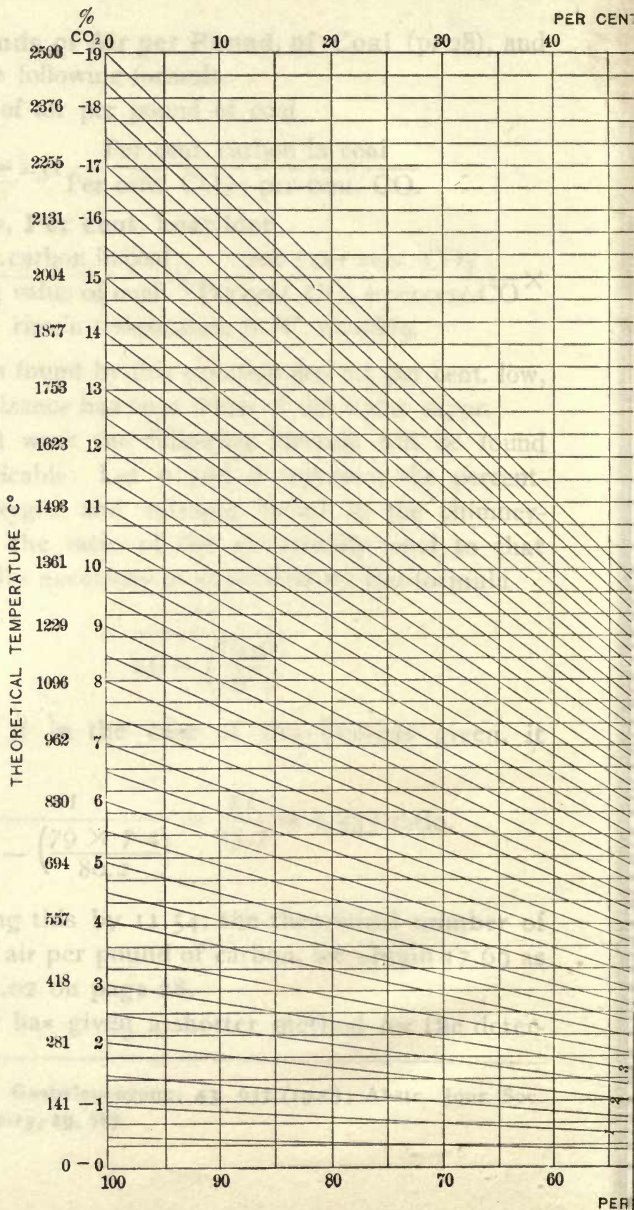
THE THERMOCHEMICAL EQUATION



PRESSURE

TAB  
 BUNTE'S CHART SHOWING HEAT LOST IN CHIM  
 TEMPE  
 PER CENT

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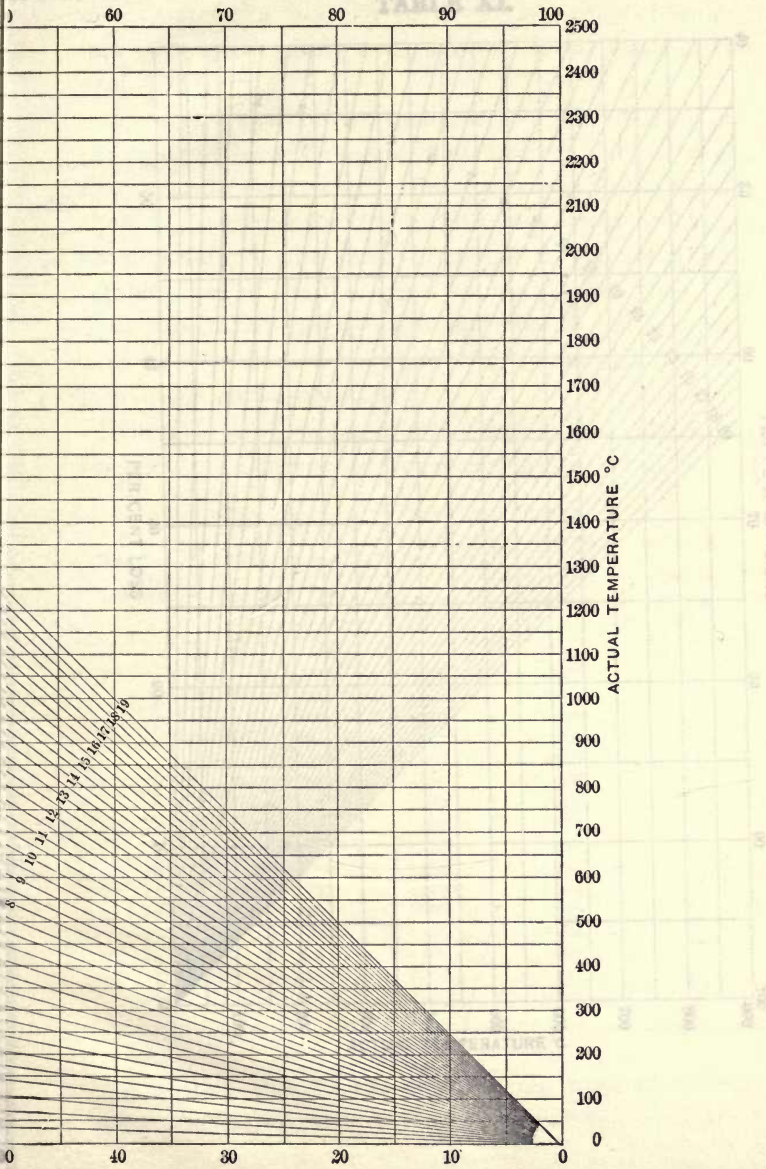




KEY-GASES FROM THE CARBONIC ACID AND THE  
TURE.

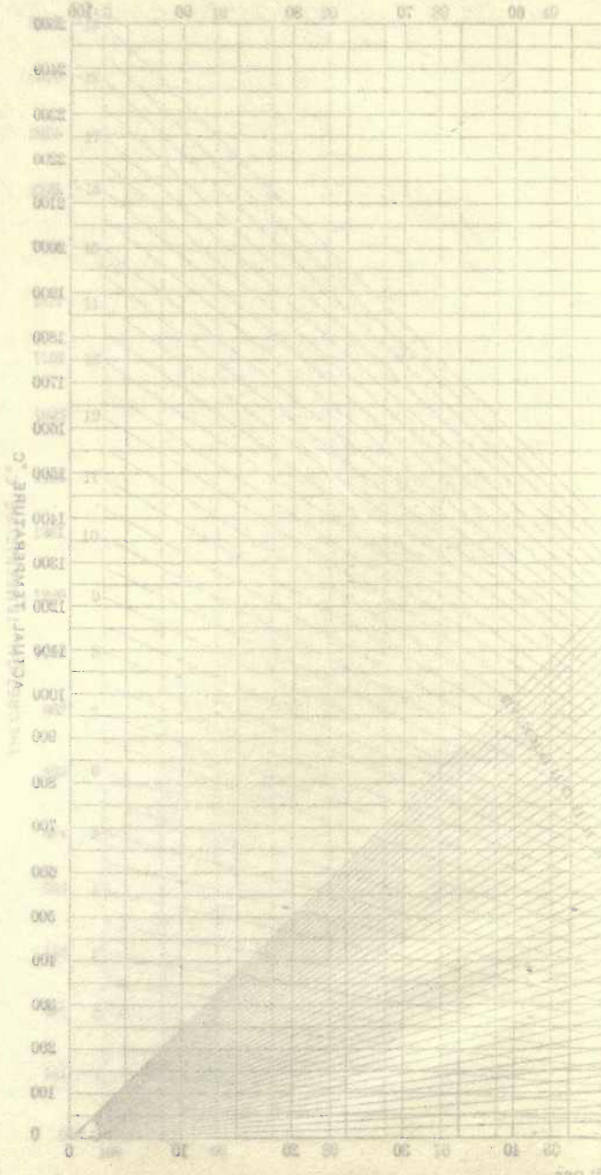
EFFICIENCY

TABLE XI.

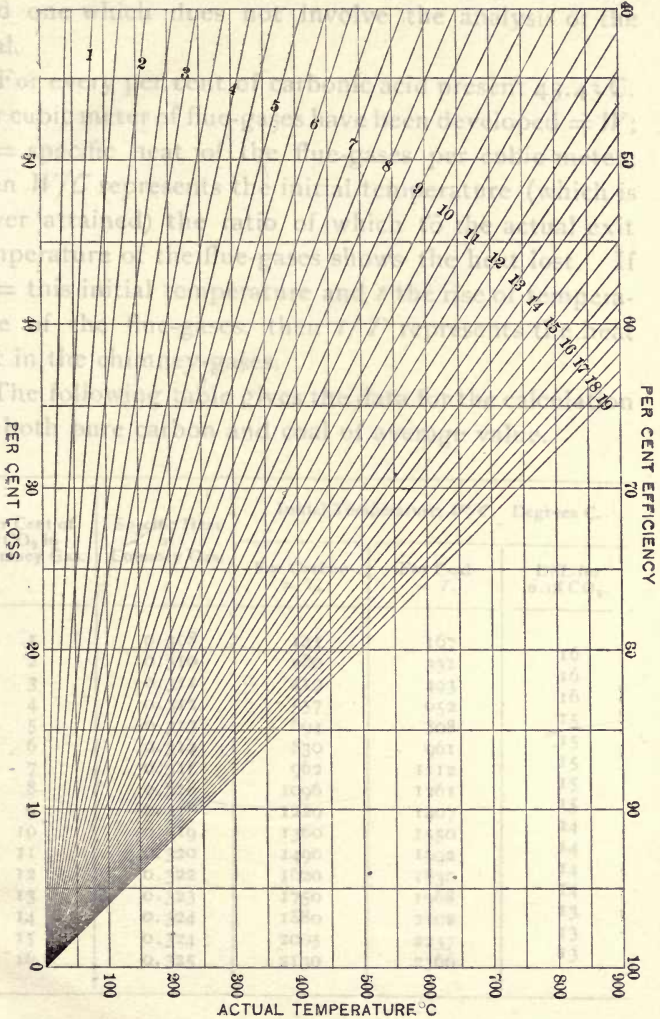


PERCENT LOSS

To face page 33.



**TABLE XI.**



Applying this to the problem on page 29 we find the initial temperature  $T$  to be  $1762^{\circ} C.$ , the rise of







mination of the quantity of heat passing up chimney, and one which does not involve the analysis of the coal.

For every per cent of carbonic acid present 43.43 C. per cubic meter of flue-gases have been developed =  $W$ ;  $C$  = specific heat of the flue-gases per cubic meter; then  $W/C$  represents the initial temperature (which is never attained) the ratio of which to the actual exit temperature of the flue-gases shows the heat lost. If  $T$  = this initial temperature and  $t$  the rise of temperature of the flue-gases, then  $t/T$  represents the heat lost in the chimney-gases.

The following table gives the data for the calculation for both pure carbon and coal of average value.

Per Cent of CO <sub>2</sub> in Chimney Gas.	Specific Heat of Chimney Gas.	Initial Temperature, $W/C$ . Degrees C.		
		For Carbon = $T$ .	For Coal = $T$ .	Diff. for 0.1% CO <sub>2</sub> .
1	0.308	141	167	
2	0.310	280	331	16
3	0.311	419	493	16
4	0.312	557	652	16
5	0.313	694	808	15
6	0.314	830	961	15
7	0.315	962	1112	15
8	0.316	1096	1261	15
9	0.318	1229	1407	15
10	0.319	1360	1550	14
11	0.320	1490	1692	14
12	0.322	1620	1830	14
13	0.323	1750	1968	14
14	0.324	1880	2102	13
15	0.324	2005	2237	13
16	0.325	2130	2366	13

Applying this to the problem on page 29 we find the initial temperature  $T$  to be 1762° C., the rise of

temperature of the gases was  $250^{\circ}\text{C.}$ , the loss is  $250/1762 = 14.2\%$ , against  $14.1\%$  found by the calculation page 31.

Bunte also employs a partially graphical method for the determination of the loss of heat. In Table X the extreme left-hand column represents the temperatures which should be obtained by the combustion of the average coal with the formation of a chimney-gas containing the percentages of carbon dioxide in the column next it. Applying this to our case we find the theoretical temperature for  $11.5\% \text{CO}_2$  to be  $1558^{\circ}$ ; dividing the rise of temperature actually observed— $250^{\circ}$ —by this, we obtain  $16.05\%$ , or  $2\%$  more than by the method of page 31.

Almost the identical result can be obtained from Table XI directly: if the point of intersection of the diagonal representing the per cent of carbon dioxide with the horizontal line denoting the actual temperature, on the right, be followed to the bottom of the table the per cent of loss is ascertained.

Table XI is the lower right-hand corner of Table X enlarged.

W. A. Noyes\* states that the following formula gives close results and is also independent of the composition of the coal.

$$\text{Percentage loss} = \left( 0.011 + \frac{100 - \% \text{CO}_2}{\% \text{CO}_2} 0.00605 \right) (t' - t).$$

Lunge† has also given a shorter method for the calculation of the heat lost.

\* Am. Chem. Journal, 19, 162.

† Zeit. f. angewandte Chemie, 1889, 240.

The following table\* shows roughly the excess of air and the per cent of heat lost in the chimney gases:

## PER CENT OF CARBONIC ACID.

2	3	4	5	6	7	8	9	10	11	12	13	14	15
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## VOLUME OF AIR MORE THAN THEORY.

(Theory = 1.0).

9.5	6.3	4.7	3.8	3.2	2.7	2.4	2.1	1.9	1.7	1.6	1.5	1.4	1.3
-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----

## PER CENT LOSS OF HEAT.

Temp. of chimney gases, 518° F.

90	60	45	36	30	26	23	20	18	16	15	14	13	12
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**Determination of Loss Due to Formation of Carbonic Oxide.**—On page 29 we see that 58 grams of carbon burned to carbonic oxide; for every gram of carbon burned to carbonic oxide there is a loss of 5.66 C., in this case a loss of 328 C. The heating value of the coal is 7220 C., hence the loss is 4.5 per cent.

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\* Arndt's Econometer Circular.

## CHAPTER V.

### APPARATUS FOR THE ANALYSIS OF FUEL AND ILLUMINATING GASES.

#### HEMPEL'S APPARATUS.

**Description.**—The apparatus, Figs. 12 and 13, is very similar in principle to that of Orsat; the burette is longer, admitting of the reading of small quantities of gas, and the pipettes are separate and mounted in brass clamps on iron stands. *P* shows a "simple" pipette \* provided with a rubber bag; this form, after ten years of use, can be said to satisfactorily take the place of the cumbersome "compound" pipette.

The pipette for fuming sulphuric acid † is shown at *F*, and differs from the ordinary in that vertical tubes after the manner of those in the Orsat pipettes replace the usual glass beads. This prevents the trapping of any gas by the filling, which was so common with the beads and glass wool. *E* represents the large explosion pipette, ‡ of about 250 cc. capacity, with walls half an inch thick; the explosion wires enter at the top and bottom to prevent short-circuiting; mercury is the confining liquid. The small explosion pipette holds

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\* Gill, Am. Chem. J., 14, 231 (1892).

† *Id.*, J. Am. Chem. Soc., 18, 67 (1896).

‡ Gill, J. Am. Chem. Soc., 17, 771 (1895).



about 110 cc. and is of glass, the same thickness as the simple pipettes. Water is here used as the confining liquid, and also usually in the burette.

An induction coil capable of giving a half-inch spark,

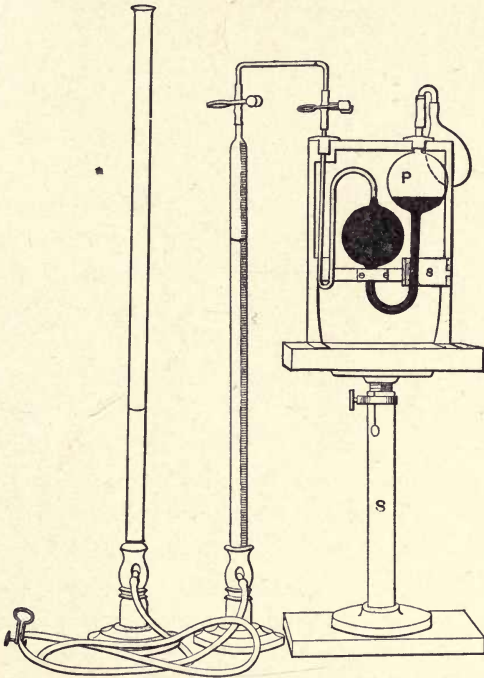


FIG. 12.—SHOWING HEMPEL BURETTE CONNECTED WITH THE SIMPLE PIPETTE ON THE STAND.

with a six-cell "Samson" battery, four "simple" pipettes and a mercury burette, complete the outfit.

The burette should be carefully calibrated and the corrections may very well be etched upon it opposite the 10-cc. divisions.

In working with the apparatus the pipettes are placed upon the adjustable stand *S* and connection made with the doubly bent capillary tube.

**Manipulation.**—To acquire facility with the use of the apparatus before proceeding to the analysis of

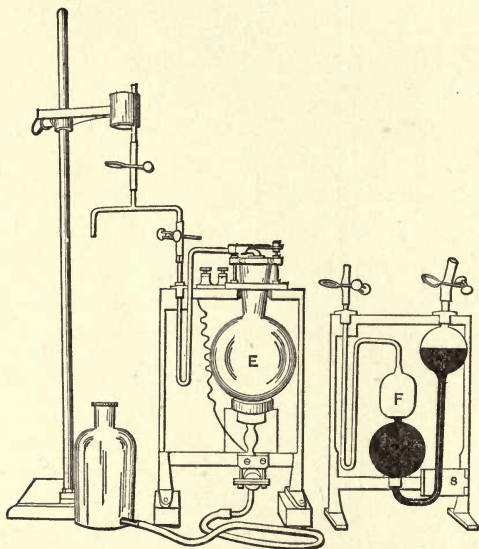


FIG. 13.—EXPLOSION PIPETTE FOR MERCURY AND SULPHURIC ACID PIPETTE.

illuminating-gas, it is well to make the following determinations, obtaining "check readings" in every case:

- I. Oxygen in air, by (1) absorption with phosphorus;
- (2) absorption with potassium (or sodium) pyrogallate;
- (3) by explosion with hydrogen.

## I. DETERMINATION OF OXYGEN IN AIR.

(1) **By Phosphorus.**—100 cc. of air are measured out as with the Orsat apparatus, the burette being allowed to drain two minutes. The rubber connectors upon the burette and pipette are filled with water, the capillary tube inserted, as far as it will go, by a twisting motion, into the connector upon the burette, thus filling the capillary with water; the free end of the capillary is inserted into the pipette connector, the latter pinched so as to form a channel for the water contained in it to escape, and the capillary twisted and forced down to the pinch-cock. There should be as little free space as possible between the capillaries and the pinch-cock. Before using a pipette, its connector (and rubber bag) should be carefully examined for leaks, especially in the former, and if any found the faulty piece replaced.

The pinch-cocks on the burette and pipette are now opened, the air forced over into the phosphorus, and the pinch-cock on the pipette closed; action immediately ensues, shown by the white fumes; after allowing it to stand for fifteen minutes the residue is drawn back into the burette, the latter allowed to drain and the reading taken. The absorption goes on best at  $20^{\circ}$  C., not at all at below  $15^{\circ}$  C.; it is very much retarded by small amounts of ethene and ammonia. No cognizance need be taken of the fog of oxides of phosphorus.

(2) **By Pyrogallate of Potassium.**—100 cc. of air are measured out as before, the carbon dioxide absorbed with potassium hydrate and the oxygen with potassium.

pyrogallate, as with the Orsat apparatus; before setting aside the pyrogallate pipette, the number of cubic centimeters of oxygen absorbed should be noted upon the slate *s* on the stand. This must never be omitted with any pipette save possibly that for potassium hydrate, as failure to do this may result in the ruin of an important analysis. The reason for the omission in this case is found in the large absorption capacity—four to five litres of carbon dioxide—of the reagent.

(3) **By Explosion with Hydrogen.**—43 cc. of air and 57 cc. of hydrogen are measured out, passed into the small explosion pipette, the capillary of the pipette filled with water, the pinch-cocks and glass stop-cock all closed, a heavy glass or fine wire gauze screen placed between the pipette and the operator, the spark passed between the spark wires, and the contraction in volume noted. *The screen should never be omitted, as serious accidents may occur thereby.* The oxygen is represented by one third of the contraction. For very accurate work the sum of the combustible gases should be but one sixth that of the non-combustible gases, otherwise some nitrogen will burn and high results will be obtained;\* that is,  $(H + O) : (N + H) :: 1 : 6$ .

## II. ANALYSIS OF ILLUMINATING-GAS.

100 cc. of gas are measured from the bottle containing the sample into the burette.

**Determination of Carbon Dioxide.**—The burette is connected with the pipette containing potassium

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\* This is shown in the work of Gill and Hunt, *J. Am. Chem. Soc.*, **17**, 987 (1895).



hydrate and the gas passed into it with shaking until no further diminution in volume takes place.

**Illuminants,  $C_nH_{2n}$ ,  $C_nH_{2n-6}$  Series.**—The rubber connectors are carefully dried out with filter-paper, a dry capillary used, and the gas passed into the pipette containing fuming sulphuric acid and allowed to stand, with occasional passes to and fro, for forty-five minutes. On account of the extremely corrosive nature of the absorbent it is not advisable to shake the pipette, as in case of breakage a serious accident might occur. For Boston gas this is sufficient, although with richer gases check readings to 0.2 cc. should be obtained. It is then passed into potassium hydrate, as in the previous determination, to remove any sulphurous acid which may have been formed and any sulphuric anhydride vapor, these having a higher vapor tension than water. The difference between this last reading and that after the absorption of the carbon dioxide represents the volume of "illuminants" or "heavy hydrocarbons" present.

As has already been stated, page 23, saturated bromine water may replace the fuming sulphuric acid. Fuming nitric acid is not recommended, as it is liable to oxidize carbonic oxide.

**Oxygen.**—This is absorbed, as in the analysis of air, by potassium or sodium pyrogallate.

**Carbonic Oxide.**—The gas is now passed into ammoniacal cuprous chloride, until the reading is constant to 0.2 cc.; it is then passed into a second pipette, which is fresh, and absorption continued until constant readings are obtained.

Gautier and Clausmann\* have shown that some carbonic oxide escapes solution in cuprous chloride, so that for very accurate work it may be necessary to pass the gas through a U-tube containing iodic anhydride heated to 70° C.

This is done by interposing this tube between the burette and a simple pipette filled with potassium hydrate. The reaction is  $5\text{CO} + \text{I}_2\text{O}_5 = 5\text{CO}_2 + 2\text{I}$ . The diminution in volume represents directly the volume of carbonic oxide present.

The volume of air contained in the tube should be corrected for as follows: One end of the tube is plugged tightly and the other end connected with the gas burette partly filled with air. A bath of water at 9° C. is placed around the U-tube and the reading of the air in the gas burette recorded when constant; the bath is now heated to 100° and the burette reading again recorded when constant. The increase in reading represents one third the volume of the U-tube,  $273:273 + (100 - 9)::3:4$ .

**Methane and Hydrogen.**—(a) *Hinman's Method.*†—The gas left from the absorption of carbonic oxide is passed into the large explosion pipette. About half the requisite quantity of oxygen (40 cc.) necessary to burn the gas is now added, mercury introduced through the T in the connector sufficient to seal the capillary of the explosion pipette, all rubber connectors carefully wired, the pinch-cocks closed, and the pipette cautiously shaken. A screen of heavy glass or fine wire gauze is interposed between the operator and the

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\* Bull. Soc. Chem. 35, 513; Abstr. Analyst, 31, 349 (1906).

† Gill and Hunt, J. Am. Chem. Soc., 17, 987 (1895).

apparatus, the explosion wires are connected with the induction coil, a spark passed between them and the pinch-cocks opened, sucking in the remainder of the oxygen. The capillary is again sealed with mercury, the stop-cock opened and closed, to bring the contents of the pipette to atmospheric pressure, and the explosion repeated as before, and the stop-cock opened.

It may be found expedient, to increase the inflammability of the mixture, to introduce 5 cc. of "detonating-gas," the hydrolytic mixture of hydrogen and oxygen. The gas in the pipette containing carbon dioxide, oxygen, and nitrogen is transferred to the mercury burette and accurately measured. The carbon dioxide resulting from the combustion of the marsh-gas is determined by absorption in potassium hydrate; to show the presence of an excess of oxygen, the amount remaining is determined by absorption with potassium pyrogallate.

The calculation is given on page 44. For very accurate work a second analysis should be made, making successive explosions, using the percentages of methane and hydrogen just found as a basis upon which to calculate the quantity of oxygen to be added each time. The explosive mixture should be so proportioned that the ratio of combustible gas (i.e.,  $\text{CH}_4$ , H and O) is to the gases which do not burn (i.e., N and the excess of  $\text{CH}_4$  and H) as 100 is to about 50 (from 26 to 64);\* otherwise the heat developed is so great as to produce oxides of nitrogen, which, being absorbed

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\* Bunsen, *Gasometrische Methoden*, 2d ed., p. 73 (1877).

in the potassium hydrate, would affect the determination of both the methane and the hydrogen. The oxygen should preferably be pure, although commercial oxygen, the purity of which is known, can be used; the oxygen content of the latter should be tested from time to time, especially with different samples.

(b) *Hempel's Method*.\*—From 12 to 15 cc. of the gas are measured off into the burette (e.g., 13.2 cc.) and the residue is passed into the cuprous chloride pipette for safe keeping. That in the burette is now passed into the small explosion pipette; a volume of air more than sufficient to burn the gas, usually about 85 cc., is accurately measured and also passed into the explosion pipette, and in so doing water from the burette is allowed to partially fill the capillary of the pipette and act as a seal. The rubber connectors upon the capillaries of the burette and pipette are carefully wired on, both pinch-cocks shut, and the stop-cock closed. The pipette is cautiously shaken, the screen interposed, the explosion wires connected with the induction coil, a spark passed between them, and the stop-cock immediately opened. The gas in the pipette, containing carbon dioxide, oxygen, and nitrogen, is transferred to the burette, accurately measured, by reading immediately, to prevent the absorption of carbon dioxide, and carbon dioxide and oxygen determined in the usual way.

**Calculation.**—(a) *Hinman's Method*.—56.2 cc. of gas remained after the absorptions; 77.4 cc. of oxygen were introduced, giving a total volume of 133.6 cc.

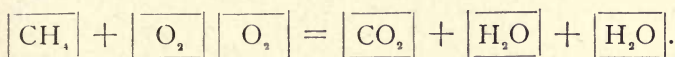
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\* Hempel, *Gas Analytische Methoden*, 3d ed., p. 245 (1901).



Residue after explosion.....	46.9 cc.
Residue after CO <sub>2</sub> absorption.....	28.2 "
Carbon dioxide formed.....	18.7 "
Contraction.....	133.6 - 46.9 = 86.7 "
Residue after O absorption.....	25.6 "
Oxygen in excess, 28.2 - 25.6 =	2.6 "

The explosion of marsh-gas or methane is represented by the equation\*

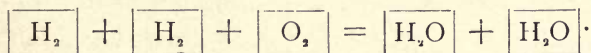


From this it is evident that the volume of carbon dioxide is equal to the volume of methane present; therefore in the above example, in the 56.2 cc. of gas burned there were 18.7 cc. methane.

The total contraction is due (1) to the disappearance of oxygen in combining with the hydrogen of the methane, and (2) to the union of the free hydrogen with oxygen. The volume of the methane having been found, (1) can be ascertained from the equation above, equals twice the volume of the methane; hence

$$86.7 - (2 \times 18.7) = 49.3 \text{ cc.},$$

contraction which is due to the combustion of hydrogen. This takes place according to the following reaction: \*




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\* H<sub>2</sub>O being as steam at 100° C. At ordinary temperatures this is condensed, giving rise to "total contraction."

Hydrogen then requires for its combustion half its volume of oxygen, hence this 49.3 cc. represents a volume of hydrogen with  $\frac{1}{2}$  its volume of oxygen, or  $\frac{3}{2}$  volumes; hence the volume of hydrogen is 32.9 cc.

(b) *Hempel's Method*.—Of the 82 cc. of gas remaining after the absorptions, 13.2 cc. were used for the explosion; 86.4 cc. air introduced giving a total volume of 99.6 cc.

Residue after explosion.....	78.0 cc.
Residue after CO <sub>2</sub> absorption.....	73.2 “
Carbon dioxide formed.....	4.8 “
Contraction.....	99.6 — 78.0 = 21.6 “
Residue after O absorption.....	70.2 “
Oxygen in excess..	73.2 — 70.2 = 3.0 “

The carbon dioxide being equal to the methane present, in the 13.2 cc. of gas burned, there were 4.8 cc. of methane. The volume of methane is found by the proportion  $13.2 : 82 :: 4.8 : x$ , whence  $x = 29.8$  cc.

The hydrogen is calculated similarly.

The following method of calculation may be substituted for that on page 43: Let  $m$  = methane,  $h$  = hydrogen,  $c$  = total contraction, and  $O$  = oxygen actually used; then

$$2m + \frac{h}{2} = O$$

and

$$2m + \frac{3h}{2} = c,$$

whence

$$m = \frac{3O - c}{4}$$

and

$$h = c - O.$$

The explosion can also be made after the absorption of oxygen and thus the troublesome absorption of carbonic oxide avoided. The calculation is then, if  $C =$  carbonic oxide,  $K = CO_2$ , formed:

$$c = \frac{C}{2} + 2m + \frac{3h}{2}, \quad . . . . . (1)$$

$$K = C + m, \quad . . . . . (2)$$

$$V = C + m + h; \quad . . . . . (3)$$

whence

$$h = V - K,$$

$$C = \frac{K}{3} + V - \frac{2c}{3},$$

$$m = \frac{2K}{3} - V + \frac{2c}{3}.$$

Another method for the estimation of hydrogen is by absorption with palladium sponge;\* it, however, must be carefully prepared, and it is the author's experience that one cannot be sure of its efficacy when it is desired to make use of it. A still better absorbent of hydrogen† is a 1 per cent solution of palladous

\* Hempel, *Berichte deutsch. ch. Gesell.*, **12**, 636 and 1006 (1879).

† Campbell and Hart, *Am. Chem. J.*, **18**, 294 (1896).

chloride at 50° C.; when fresh this will absorb 20–50 cc. of hydrogen in ninety minutes. A proportionately longer time is required if more hydrogen be present or the solution nearly saturated. The methane could then be determined by explosion or by mixing with air and passing to and fro over a white-hot platinum spiral in a tubulated pipette called the grisometer\* (grisou = methane).

**Nitrogen.**—There being no direct and convenient method for its estimation with this apparatus, the percentage is obtained by finding the difference between the sum of all the percentages of the gases determined and 100 per cent.

New † determines nitrogen in illuminating-gas directly after the method of Dumas in organic substances; 150 cc. of gas are used, the hydrocarbons partially absorbed by fuming sulphuric acid and the remainder burned in a combustion tube with copper oxide; the carbon dioxide is absorbed and the residual nitrogen collected and measured.

**Accuracy and Time Required.**—For the absorptions the apparatus is accurate to 0.1 cc.; for explosions by Hinman's method ‡ the methane can be determined within 0.2 per cent, the hydrogen within 0.3 per cent; by Hempel's method within 1 per cent for the methane and 7.5 per cent for the hydrogen. The time required for the analysis of illuminating-gas is from three to three and one-half hours; for air, from fifteen to twenty minutes.

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\* Winkler, *Fres. Zeit.*, 28, 269 and 288.

† *J. Soc. Chem. Ind.*, 11, 415 (1892).

‡ Gill and Hunt, *loc cit.*



*Notes.*—The object in filling the capillaries of the explosion pipettes with water or mercury before the explosion is to prevent the bursting of the rubber connectors on them. With mercury this is effected by introducing it through the T joint in the connector. After testing for oxygen with the pyrogallate a small quantity of dilute acetic acid is sucked into the burette to neutralize any alkali which by any chance may have been sucked over into it. The acid is rinsed out with water and this forced out by mercury before the burette is used again.

The water in the burette should be saturated with the gas which is to be analyzed—as illuminating-gas—before beginning an analysis. The reagents in the pipettes should also be saturated with the gases for which they are not the reagent. For example, the fuming sulphuric acid should be saturated with oxygen, carbon monoxide, methane, hydrogen, and nitrogen; this is effected by making a blank analysis using illuminating-gas.

The method of analysis of the residue after the absorptions have been made by explosion is open to two objections: 1st, the danger of burning nitrogen by the violence of the explosion; and 2d, the danger of breakage of the apparatus and possible injury to the operator. These may be obviated by employing the apparatus of Dennis and Hopkins,\* which is practically a grisometer with mercury as the confining liquid; or that of Jager,† who burns the gases with oxygen in a

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\* J. Am. Chem. Soc., 21, 398 (1899).

† J. f. Gasbeleuchtung, 41, 764. Abstr. J. Soc. Chem. Ind., 17, 1190 (1898).

hard-glass tube filled with copper oxide. By heating to  $250^{\circ}$  C. nothing but hydrogen is burned; higher heating of the residue burns the methane. Or the mixture of oxygen and combustible gases, bearing in mind the ratio mentioned at the bottom of page 43; can be passed to and fro through Drehschmidt's\* capillary heated to bright redness. This consists of a platinum tube 20 cm. long, 2 mm. thick, 1.7 mm. bore, filled with three platinum or palladium wires. The ends of the tube are soldered to capillary brass tubes and arranged so that these can be water cooled. It is inserted between the burette and a simple pipette, mercury being the confining liquid in both cases. The air contained in the tube can be determined as in the case of the tube containing iodic anhydride, p. 42.

To the method of explosion by the mixture of an aliquot part of the residue with air, method (b), there is the objection that the carbon dioxide formed is measured over water in a moist burette, giving abundant opportunities for its absorption, and that the errors in analysis are multiplied by about six, in the example by  $\frac{8\frac{2}{3}}{1\frac{3}{2}}$ .

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\* Ber. d. deut. chem. Gesell. 21, 3242 (1888).

## CHAPTER VI.

### REAGENTS AND ARRANGEMENT OF THE LABORATORY.

THE reagents used in gas-analysis are comparatively few and easily prepared.

**Hydrochloric Acid**, Sp. gr. 1.10.—Dilute “muriatic acid” with an equal volume of water. In addition to its use for preparing cuprous chloride, it finds employment in neutralizing the caustic solutions which are unavoidably more or less spilled during their use.

**Fuming Sulphuric Acid**.—Saturate “Nordhausen oil of vitriol” with sulphuric anhydride. Ordinary sulphuric acid may be used instead of the Nordhausen; in this case about an equal weight of sulphuric anhydride will be necessary. *Absorption capacity*, 1 cc. absorbs 8 cc. of ethene (ethylene).

**Acid Cuprous Chloride**.—The directions given in the various text-books being troublesome to execute, the following method, which is simpler, has been found to give equally good results. Cover the bottom of a two-liter bottle with a layer of copper oxide or “scale”  $\frac{3}{8}$  in. deep, place in the bottle a number of pieces of rather stout copper wire reaching *from top to bottom*, sufficient to make a bundle an inch in diameter, and fill the bottle with common hydrochloric

acid of 1.10 sp. gr. The bottle is occasionally shaken, and when the solution is colorless, or nearly so, it is poured into the half-liter reagent bottles, containing copper wire, ready for use. The space left in the stock bottle should be immediately filled with hydrochloric acid (1.10 sp. gr.).

By thus adding acid or copper wire and copper oxide when either is exhausted, a constant supply of this reagent may be kept on hand.

*The absorption capacity* of the reagent per cc. is, according to Winkler, 15 cc. CO; according to Hempel 4 cc. The author's experience with Orsat's apparatus gave 1 cc.

Care should be taken that the copper wire does not become entirely dissolved and that it extend from the top to the bottom of the bottle; furthermore the stopper should be kept thoroughly greased the more effectually to keep out the air, which turns the solution brown and weakens it.

**Ammoniacal Cuprous Chloride.** — The acid cuprous chloride is treated with ammonia until a faint odor of ammonia is perceptible; copper wire should be kept in it similarly to the acid solution. This alkaline solution has the advantage that it can be used when traces of hydrochloric acid vapors might be harmful to the subsequent determinations, as, for example, in the determination of hydrogen by absorption with palladium. It has the further advantage of not soiling mercury as does the acid reagent.

*Absorption capacity*, 1 cc. absorbs 1 cc. CO.

Cuprous chloride is at best a poor reagent for the absorption of carbonic oxide; to obtain the greatest



accuracy where the reagent has been much used, the gas should be passed into a fresh pipette for final absorption, and the operation continued until two consecutive readings agree exactly. The compound formed by the absorption—possibly  $\text{Cu}_2\text{COCl}_2$ —is very unstable, as carbonic oxide may be freed from the solution by boiling or placing it in vacuo; even if it be shaken up with air, the gas is given off, as shown by the increase in volume and subsequent diminution when shaken with fresh cuprous chloride.

**Hydrogen.**—A simple and effective hydrogen generator can be made by joining two six-inch calcium chloride jars by their tubulatures. Pure zinc is filled in as far as the constriction in one, and the mouth closed with a rubber stopper carrying a capillary tube and a pinch-cock. The other jar is filled with sulphuric acid 1 : 5 which has been boiled and cooled out of access of air. The mouth of this jar is closed with a rubber stopper carrying one of the rubber bags used on the simple pipettes.

**Mercury.**—The mercury used in gas analysis should be of sufficient purity as not to “drag a tail” when poured out from a clean vessel. It may perhaps be most conveniently cleaned by the method of J. M. Crafts, which consists in drawing a moderate stream of air through the mercury contained in a tube about 3 feet long and  $1\frac{1}{4}$  inches internal diameter. The tube is supported in a mercury-tight V-shaped trough, of size sufficient to contain the metal if the tube breaks, one end being about 3 inches higher than the other. Forty-eight hours’ passage of air is sufficient to purify any ordinary amalgam. The mercury may very well

be kept in a large separatory funnel under a layer of strong sulphuric acid.

**Palladous Chloride.**—5 grams palladium wire are dissolved in a mixture of 30 cc. hydrochloric and 2 cc. nitric acid, this evaporated just to dryness on a water-bath, re-dissolved in 5 cc. hydrochloric acid and 25 cc. water, and warmed until solution is complete. It is diluted to 750 cc. and contains about one per cent of palladous chloride. It will absorb about two thirds of its volume of hydrogen.

**Phosphorus.**—Use the ordinary white phosphorus cast in sticks of a size suitable to pass through the opening of the tubulated pipette.

**Potassium Hydrate.**—(a) For carbon dioxide determination, 500 grams of the commercial hydrate is dissolved in 1 liter of water.

*Absorption capacity*, 1 cc. absorbs 40 cc. CO<sub>2</sub>.

(b) For the preparation of potassium pyrogallate for special work, 120 grams of the commercial hydrate is dissolved in 100 cc. of water.

**Potassium Pyrogallate.**—Except for use with the Orsat or Hempel apparatus, this solution should be prepared only when wanted. The most convenient method is to weigh out 5 grams of the solid acid upon a paper, pour it into a funnel inserted in the reagent bottle, and pour upon it 100 cc. of potassium hydrate (a) or (b). The acid dissolves at once, and the solution is ready for use.

If the percentage of oxygen in the mixture does not exceed 28, solution (a) may be used;\* if this amount be exceeded, (b) must be employed. Otherwise carbonic oxide may be given off even to the extent of 6 per cent.

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\* Clowes, Jour. Soc. Chem. Industry, 15, 170.

Attention is called to the fact that the use of potassium hydrate purified by alcohol has given rise to erroneous results.

*Absorption capacity*, 1 cc. absorbs 2 cc. O.

**Sodium Hydrate.**—Dissolve the commercial hydrate in three times its weight of water. This may be employed in all cases where solution (*a*) of potassium hydrate is used. The chief advantage in its use is its cheapness, it costing but one tenth as much as potassium hydrate, a point to be considered where large classes are instructed. Sodium pyrogallate is, however, a trifle slower in action than the corresponding potassium salt.

#### ARRANGEMENT OF THE LABORATORY.

The room selected for a laboratory for gas-analysis should be well lighted, preferably from the north and east. To prevent changes in temperature it should be provided with double windows, and the method of heating should be that which will give as equable a temperature as possible. In the author's laboratory, instead of the usual tables, shelves are used, 18 inches wide and  $1\frac{1}{4}$  inches thick, best of slate or soapstone, firmly fastened to the walls, 30 inches from the floor; the Orsat apparatus, when not in use, may be suspended from these. The reagents are contained in half-liter bottles fitted with rubber stoppers, placed upon a central table convenient to all. Here are found scales, funnels and graduates for use in making up reagents. Distilled water is piped around to each place by  $\frac{1}{8}$ -inch tin pipe and  $\frac{3}{16}$ -inch rubber tubing from a  $\frac{1}{4}$ -inch "main," being supplied at the tem-

perature of the room from bottles placed about six feet above the laboratory shelves. A supply of a gallon per day per student should be provided.

At the right of each place is fixed a sand-glass of cylindrical rather than conical form, graduated to minutes for the draining of the burettes. The "egg-timers" found in kitchen-furnishing stores serve the purpose admirably.

"Unknown gases" for analysis are best contained in a Muencke double aspirator, Fig. 14, where they

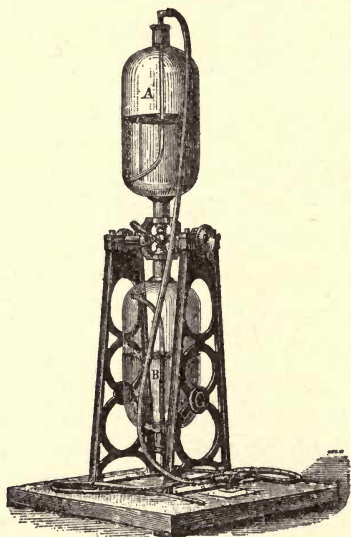


FIG. 14.—MUECKE'S ASPIRATOR.

can be thoroughly mixed before distribution and conveyed by a pipe to the central table.

Finally, the laboratory should contain a stone-ware sink provided with an efficient trap of the same



material, to prevent mercury from being carried into and corroding the lead waste-pipes.

Drawers should be provided with compartments for various sizes of rubber connectors, pinchcocks, glass tubing, stoppers and fittings, and tools. When working with the Orsat apparatus alone, three feet of shelf space may be allowed to each student; when using this with another, as, for example, the Bunte, another foot should be added.

The course which the writer has been in the habit of giving to the Mechanical and Electrical Engineers embraces two exercises in the laboratory of two hours each, supplemented with four hours of lectures. The students in the laboratory make an analysis of air and an "unknown" furnace-gas, take and analyze an actual sample of chimney-gas, and make the calculation of heat lost and air used. In the lectures, the subject of gas-analysis and its other applications, and of fuels, their origin, description, preparation, analysis, and determination of heating value, are described.

## CHAPTER VII.

### FUELS—SOLID, LIQUID, AND GASEOUS: THEIR DERIVATION AND COMPOSITION.

The substances employed as fuels are:

*a.* SOLID FUELS.—Wood, peat, brown, bituminous and anthracite coal, charcoal, coke, and oftentimes various waste products, as sawdust, bagasse, straw, and spent tan.

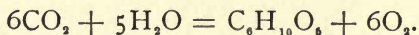
*b.* LIQUID FUELS.—Crude petroleum and various tarry residues.

*c.* GASEOUS FUELS.—Natural gas, producer, blast-furnace, water, and illuminating gas.

The essential constituents in all these are carbon and hydrogen; the accessory, oxygen, nitrogen, and ash; and the deleterious, water, sulphur, and phosphorus.

#### *a.* SOLID FUELS.

**Wood** is composed of three substances—*cellulose*, or woody fibre ( $C_6H_{10}O_6$ )<sub>n</sub>; *the components of the sap*, the chief of which is lignine, a resinous substance of identical formula with cellulose; and *water*. The formation of cellulose from carbon dioxide and water may be represented by the equation



The amount of water which wood contains determines its value as a fuel. This varies from 29 per cent in ash

to 50 per cent in poplar; it varies also with the season at which the wood is cut, being least when the sap is in the roots—in December and January. This difference may amount to 10 per cent in the same kind of wood.

The harder varieties of wood make the best fuel, a cord of seasoned hardwood being about equal to a ton of coal. Yellow pine, however, has but half this value; the usual allowance in a boiler-test is 0.4 the value of an equal weight of coal.

The ash of wood is mainly potassium carbonate, with traces of other commonly occurring substances, as lime, magnesia, iron, silica, and phosphoric acid.

The *percentage composition of wood* may be considered as approximately,

Water.	Carbon.	Hydrogen.	Oxygen.	Ash.	Sp. Gr.
20	39	4.4	35.6	1	0.5.*

When burned it yields about 4000 C. per kilo, and requires 6 times its weight of air or 4.6 cu. m. (74.1 cu. ft. per pound) for its combustion.

**Peat** finds considerable application in Europe, and is coming into use in this country in the form of briquettes. To this end it is reduced to a dry powder and compressed into small cylindrical blocks; it is claimed to be as efficient as coal at half the price. It is also proposed to gasify peat after the manner of coal. Peat is produced by the slow decay under water of certain swamp plants, more especially the mosses (Sphagnaceæ), evolving methane ( $\text{CH}_4$ ) (marsh-gas) and carbon dioxide ( $\text{CO}_2$ ).

It contains considerable moisture, from 20 to 50 per cent, and 10 per cent even when "thoroughly

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\* Mills & Rowan, Fuels, p. 11.

dry." Thirty per cent of its available heat is employed in evaporating this moisture. The high content of ash, from 3 to 30 per cent, averaging 15 per cent, also diminishes its value as a fuel.

The ash of peat differs from that of wood in containing little or no potassium carbonate.

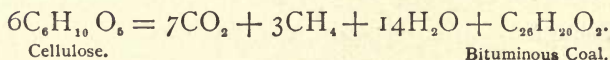
The *percentage composition of peat* may be considered as approximately,

	Water.	Carbon.	Hydrogen.	Oxygen.	Nitrogen.	Ash.	Sp. Gr.
German....	16.4	41.0	4.3	23.8	2.6	11.9	1.05
American...	20.8	40.8	4.4		26.6	7.7	—

Such peat is about equivalent to wood in its heating effect, one pound evaporating from 4.5 to 5 pounds of water.

**Coal.**—Geologists tell us that coal was probably produced by the decay under fresh water of plants belonging principally to the Conifer, Fern and Palm families; these flourished during the Carboniferous Age to an extent which they never approached before or since. Representatives of the last family, which it is thought produced most of the coal, have been found 2 to 4 feet in diameter and 80 feet in height.

By their decay, carbon dioxide "choke-damp," marsh-gas "fire-damp," and water were evolved. The change might be represented by the equation



Some idea of the density of the vegetation and the time required may be obtained from the fact that it has been calculated that 100 tons of vegetable matter—the amount produced per acre per century—if compressed to the specific gravity of coal and spread over



an acre would give a layer less than 0.6 of an inch thick. Now four fifths of this is lost in the evolution of the gaseous products, giving as a result an accumulation of *one eighth of an inch per century*, or one foot in 10,000 years.\*

**Brown Coal or Lignite** may be regarded as forming the link between wood and coal; geologically speaking it is of later date than the true coal. Most of the coal west of the Rocky Mountains is of this variety.

As its name denotes, it generally is of brown color—although the western coal is black—and has a conchoidal fracture. It contains a large quantity of water when first mined, as much as 60 per cent, and when “air-dry” from 15 to 20 per cent. The per cent of ash is also high, from 1 to 20 per cent.

The average moisture and ash in American lignites are 12.75 and 6.1 respectively.

The *percentage composition of brown coal* may be considered as approximately,

	Water.	Carbon.	Hydrogen.	Oxygen & Nitrogen.	Ash.	Sp. Gr.
German	18.0	50.9	4.6	16.3	10.2	1.3

**Bituminous Coal.**—This is the variety from which all the following coals are supposed to have been formed, by a process of natural distillation combined with pressure. According to the completeness of this process we have specimens which contain widely differing quantities of volatile matter. This forms the true basis for the distinguishing of the varieties of coal. In ordinary bituminous coal this volatile matter amounts to 30 or 40 per cent. Three varieties of bituminous coal are ordinarily distinguished, as follows:

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\* In case the student desires to follow in a more extended manner the geology of coal, reference may be had to Le Conte's "Elements of Geology." pp. 345-414, 3d ed.

*Dry or non-caking*—those which burn freely with but little smoke and—as the name denotes—do not cake together when burned. The coals from Wyoming are an example of this class.

*Caking*—those which produce some smoke and cake or sinter together in the furnace. An example of these is the New River and Connellsville coal.

*Fat or Long-flaming*—those producing much flame and smoke and do or do not cake in burning; volatile matter 50 per cent or more. Some of the Nova Scotia coals belong to this class.

Bituminous coal varies much in its composition—is black or brownish black, soft, friable, lustrous, and of specific gravity of 1.25 to 1.5.

Moisture varies from 0.25 to 8 per cent, averaging about 5.

The *percentage composition of bituminous coal* may be considered as approximately,\*

Water.	Carbon.	Hydrogen.	Oxygen.	Nitrogen	Ash.	Sulphur.
0.9	77.1	5.2	6.7	1.6	7.6	1.0
Water.	Volatile Matter.		Fixed Carbon.		Ash.	
0.9	27.4		64.1		7.6	

*Semi-Bituminous or Semi-Anthracite Coal* is upon the border-line between the preceding and the following variety; it is harder or softer than bituminous, contains less volatile matter (15 to 20 per cent), and burns with a shorter flame. An example of this is the Pocahontas coal.

The *percentage composition of semi-bituminous and semi-anthracite coal* may be considered to be approximately,\*

Water.	Carbon.	Hydrogen.	Oxygen.	Nitrogen.	Ash.	Sulphur.
0.5	83.0	4.7	4.2	1.3	5.5	0.8
Water.	Volatile Matter.		Fixed Carbon.		Ash.	
0.5	16.7		77.3		5.5	

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\* H. J. Williams.

**Anthracite Coal** is the hardest, most lustrous, and densest of all the varieties of coal, having a specific gravity of 1.3 to 1.75; it contains the most carbon and least hydrogen and volatile matter (5 to 10 per cent). It has a vitreous fracture and kindles with difficulty, burning with a feeble flame, giving little or no smoke and, with sufficient draft, an intense fire. The Lehigh coal is an excellent example of this class.

The *percentage composition of anthracite coal* may be considered as approximately,\*

Water.	Carbon.	Hydrogen.	Oxygen.	Nitrogen.	Ash.	Sulphur.
2.0	83.9	2.7	2.8	0.8	7.2	0.6
Water.	Volatile Matter.		Fixed Carbon.		Ash.	
2.0	4.3		86.5		7.2	

The ash of coal varies from 1 to 20 per cent and is mainly clay—silicate of alumina—with lime, magnesia, and iron. When coal is burned it yields from 6100 to 8000 C. and requires about 12 times its weight of air, 9.76 cu. m. per kilo or 156.7 feet per pound. For the greatest economy Scheurer-Kestner † found that this should be increased from 50 to 100 per cent.

**Charcoal** is prepared by the distillation or smouldering of wood, either in retorts, where the valuable by-products are saved, or in heaps. It should be jet-black, of bright lustre and conchoidal fracture.

When wood is charred in heaps only about 20 per cent of its weight in charcoal is obtained—48 bushels per cord, or about half the percentage of carbon. When retorts or kilns are employed, the yield is increased to 30 per cent, and 40 per cent of pyroligneous

\* H. J. Williams.

† Jour. Soc. Chem. Industry, 7, 616.

acid of 10 per cent strength, with 4 per cent of tar, are obtained.

The *percentage composition of wood-charcoal* may be considered as approximately,

Carbon.	Ash.	Sp. Gr.
97.0	3.0	0.2

**Coke** is prepared by the distillation of bituminous coal in ovens; these are of two types, those in which the distillation-products are allowed to escape—the “beehive” ovens—and those in which they are carefully saved, as the Otto-Hoffman, Semet-Solvay, Simon-Carvés’, and others.

The “beehive” ovens yield from 50 to 65 per cent of the weight of the coal—about  $2\frac{1}{2}$  tons. The Otto-Hoffman ovens are long narrow thin-walled retorts 33 by 6 by 1.5 feet,\* regeneratively heated by side and bottom flues; the charge is about 6 tons of coal, and the following percentage yields of by-products are obtained: coke 70–75, gas 16 (10 M. cu. ft.), tar 3.3–5.6, ammonia 0.3–1.4.† The Semet-Solvay ovens differ from the above in that they are not regeneratively heated and their walls are thicker, serving to store up the heat; the yield of coke is somewhat higher—about 80 per cent.‡ The by-products obtained alone increase the value of the output about one and one half times. Good coke should possess a cellular structure, a metallic ring, contain practically no impurities, and be capable of bearing a heavy burden in the furnace.

\* Irwin, Eng. Mag., Oct. 1901, abstr. J. Am. Chem. Soc., 24, 40.

† H. O. Hofman, Tech. Quar., 11, 212 (1898).

‡ Pennock, J. Am. Chem. Soc., 21, 678 (1899).



The analysis of Connellsville coke with the coal from which it is prepared is given below.

	Water.	Volatile Matter.	Carbon.	Sulphur.	Ash.
Coal	1.26	30.1	59.62	0.78	8.23
Coke	0.03	1.29	89.15	0.084	9.52
Otto-Hoffman coke:					
	3.7	1.3	Fixed Carbon. 86.1		8.9
Heating value.....					7100 C.

*The Minor Solid Fuels.*

**Sawdust** and **Spent Tan-bark** find occasional use, their value depending upon the quantity of moisture they contain. With 57 per cent of moisture 1 pound of tan-bark gave an evaporation of 4 pounds of water.

**Wheat Straw** finds application as fuel in agricultural districts,  $3\frac{1}{2}$  pounds being equal to 1 pound of coal. Upon sugar-plantations the crushed cane or **Bagasse**, partially dried, is extensively used as a fuel. With 16 per cent of moisture an evaporation of 2 pounds of water per pound of fuel has been obtained.

**Briquets, "Patent Fuel."**\*—In Europe coal dust is cemented together with some tarry binding material and baked or compressed into blocks usually about  $6 \times 2 \times 1$  inches, which form a favorite fuel for domestic purposes. In some cases they take the form and size of a large goose egg, and are called *eggettes*: these are being made, among other places, at Scranton, Pa., and withstand well the shocks incident to shipment.

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\* Condition of the Coal Briquetting Industry in the United States, E. W. Parker, Bull. No. 316 U. S. Geol. Survey, Contributions to Economic Geology, 1906. Part II, Coal Lignite and Peat, pp. 460-485.

**Storage of Coal and Spontaneous Combustion.**—

While authorities differ as to the way and manner in which coal should be stored, as regards height of pile, number, size, and arrangement of ventilating channels, they are practically agreed that it should always be covered. Six months' exposure to the weather may with European coals cause a loss of from 10 to 40 per cent in heating value, while with Illinois coals it varies from 2 to 10 per cent.\* The North German Lloyd Steamship Company stores its coal in a covered bin provided with ventilators, and restricts the height of the pile to 8 feet. A large gas company in a western city also uses a covered bin, with ventilators 8 inches square every 20 feet; the height of the pile may be from 10 to 15 feet. An electric company in the same city † has arranged to store 14,000 tons of coal under water in 12 pits, a steam-shovel being used to dig out the coal. Ventilating flues serve the additional purposes of enabling the temperature of the pile to be ascertained before ignition takes place, and as a means of introduction of either steam or carbonic acid to extinguish any fire which may occur. All the supports of the bin in contact with the coal should be of brick, concrete or iron, and if of hollow iron, filled with cement.

The spontaneous combustion of coal is due primarily to the rapid absorption of oxygen by the finely divided coal, and to the oxidation of iron pyrites, "coal brasses," occurring in the coal. The conditions favorable to the process are:

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\* Parr and Hamilton, Univ. of Ill., Bulletin 4, No. 33, August, 1907.

† Eng. and Min. Jour., September 15, 1906.

First. A supply of air sufficient to furnish oxygen, but of insufficient volume to carry off the heat generated.

Second. Finely divided coal, presenting a large surface for the absorption of oxygen.

Third. A considerable percentage of volatile matter in the coal.

Fourth. A high external temperature.

A method of extinguishing a fire in a coal pile not provided with ventilators consists in removing and spreading out the coal and flooding the burning part with water. Another method consists in driving a number of iron or steel pipes provided with "driven well points" at the place where combustion is taking place, and forcing water or steam through these upon the fire.

### *b.* LIQUID FUELS.

These consist of petroleum and its products, and various tarry residues from processes of distillation, as from petroleum, coking-ovens, wood and shale. Liquid fuel possesses the advantage that it contains no ash, is easily manipulated, the fire is of very equable temperature, very hot, and practically free from smoke.

Regarding the origin of petroleum, many theories have been proposed. That of Engler,\* that it was formed by the distillation under pressure of animal fats and oils, the nitrogenous portions of the animals previously escaping as amines, seems most probable; it has yielded the best results of any hypothesis when tested upon an industrial scale.

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\* Jour. Soc. Chem. Industry, 14, 648.

**Crude Petroleum** varies greatly in color according to the locality; it is usually yellowish, greenish, or reddish brown, of benzine-like odor, and sp. gr. of 0.78 to 0.80. It "flashes" at the ordinary temperature; hence great care should be employed in its use and storage. Its *percentage composition* is shown below.

Carbon.	Hydrogen.
84.0-85.0	16.0-15.0

It is more than twice as efficient as the best anthracite coal. In practice 14 to 16 pounds of water per pound of petroleum have been evaporated, and an efficiency of 19,000 B. T. U. was obtained as against 8500 B. T. U. for anthracite. In general  $3\frac{1}{2}$  to 4 barrels of oil are equivalent to a ton of good soft coal.\*

### c. GASEOUS FUELS.

**Natural Gas** is usually obtained when boring for petroleum and consists mainly of methane and hydrogen, although the percentage varies with the locality. The Findlay, Ohio,† gas is of the following composition:

CH <sub>4</sub>	H	N	O	C <sub>2</sub> H <sub>4</sub>	CO <sub>2</sub>	CO	H <sub>2</sub> S	Sp Gr.
92.6	2.3	3.5	0.3	0.3	0.3	0.5	0.2	0.57

**Blast-furnace, Producer, or Generator Gas** is the waste gas issuing from the top of a blast-furnace or obtained by partially burning coal by a current of air (pro-

\* W. B. Phillips, *Texas Petroleum* (1900), p. 84.

† Orton, *Geology of Ohio*, vol vi. p. 137.



duced by steam) in a special furnace—a gas-producer or generator. It is mainly carbonic oxide and nitrogen.

	CO	N	CO <sub>2</sub>	H	CH <sub>4</sub>	O	B.T.U. per Foot.
Blast-furnace gas.....	34.3	63.7	0.6	1.4	—	—	—
Gas from bitumin. coal	24.5	46.8	3.7	17.8	6.8	0.4	223
“ “ “ “	25.0	41.4	4.0	19.4	9.6	0.6	—
“ “ anthrac. “	27.0	57.3	2.5	12.0	1.2	—	—
“ “ “ “	17.2	53.1	8.6	18.2	2.4	0.4	140
“ “ “ “	26.0	47.0	8.0	18.5	0.5	—	145*

One ton of coal yields from 160† to 170 thousand cubic feet of gas of 156 to 138 B.T.U. heating power, or 81 to 86 or even 90 per cent of the value of the coal.

**Water-gas.**—If, instead of passing simply air over hot coal, water-vapor, or rather steam, be employed, it is decomposed, giving carbonic oxide and hydrogen, according to the equation  $H_2O + C = CO + H_2$ , and the resulting mixture is called water-gas. The *percentage composition*, which varies according to the apparatus and fuel employed, is about as follows:

	CO	H	CH <sub>4</sub>	CO <sub>2</sub>	N	O	Illts.	Sp. Gr.
From coke....	45.8	45.7	2.0	4.0	2.0	0.5	—	0.57
From bit. coal	34.0	41.9	7.5	5.4	9.2	1.1	0.9	—

Fischer ‡ states that 1 ton of coke gives about 36 thousand cubic feet of gas, equivalent to 42 per cent of the value of the coal. From 1 ton of bituminous coal about 51 thousand cubic feet of gas of 360 B.T.U. heating power are obtained, or an efficiency of nearly 62 per cent.§

\* Suction gas producer.

† Humphrey, Jour. Soc. Chem. Industry, 20, 107 (1901); *ibid.*, 16, 522 (1897).

‡ Taschenbuch für Feuerungs-Techniker, p. 27.

§ Slocum, J. Soc. Chem. Industry, 16, 420 (1897).

**Coal or Illuminating Gas** was formerly produced by the distillation of bituminous coal; it is at present largely made by the enriching of water-gas. "Gas-oil," a crude naphtha, is blown into the water-gas generator and changed to a permanent gas by the heat. It is of the following composition:

	H	CH <sub>4</sub>	CO	C <sub>2</sub> H <sub>4</sub>	CO <sub>2</sub>	N	O	Sp. Gr.
Coal gas.....	47.0	40.5	6.0	4.0	0.5	1.5	0.5	0.4
Enriched water-gas	27.9	25.9	25.3	15.0	2.9	3.0	0.0	0.6

One ton of coal gives about 10 thousand cubic feet of gas, or about 20 per cent of the heating value of the coal.

### *Heating Value of these Gases.*

The following table, mainly from Slocum,\* gives an idea of the comparative value of the gases:

Name of Gas.	B. T. U. per Cu. Ft.†	Yield.	Air Required for Combustion per Cu. Ft.
Oil.....	1350	77 cu. ft. per gal.	....
Natural.....	980 840-1170	....	9.80
		Thousand Ft. per Ton.	
Enriched water.....	700	40 ‡	....
Coke-oven.....	686	5	....
Coal .....	600-625	10	5.65
Blue water.....	332-500	5	....
Heating (coke-oven).	367	5	....
Bit. coal water.....	342	51	2.97
Mond producer.....	156	160	1.25
Siemens producer....	137	170	....
Wood or peat.....	140-145	....	....

\* Slocum, J. Soc. Chem. Industry, 16, 420 (1897).

† Determined with the Junkers calorimeter.

‡ 168-200 gallons of "gas-oil" are also required.

REFERENCES.—*Report of U. S. "Liquid Fuel" Board*, Dept. of Navy, Bureau of Steam Engineering, Washington, 1904. pp. 450.

*Report on the Operations of the Coal-Testing Plant of the U. S. Geological Survey at St. Louis*, 1904. Professional Paper, No. 48, Parts I, II, and III. 1906.

*Preliminary Report on the Operations of the Coal Testing Plant of the U. S. Geol. Survey at St. Louis*, 1904.

Bull. No. 261, 1905.

Bull. No. 261 for 1905.

Bull. No. 290, 1906.

*A Study of Four Hundred Steaming Tests*, made at Fuel Testing Plant at St. Louis in 1904, 1905, 1906, by L. P. Breckenridge. Bull. No. 325, U. S. Geol. Survey, 1907.

*The Burning of Coal without Smoke*. D. T. Randall. Bull. No. 334, U. S. Geol. Survey, 1908.

Barr, "*Boilers and Furnaces*."

Hodgetts, "*Liquid Fuels*."

## CHAPTER VIII.

### METHODS OF ANALYSIS AND DETERMINATION ON THE HEATING VALUE OF FUEL.

#### SAMPLING.

A FEW representative lumps or shovelfuls are taken from each barrow or from various points in the pile in boiler tests. Shovelfuls of coal should be taken at regular intervals and put into a *tight covered* barrel or some air-tight receptacle, and the latter should be placed where it is protected from the heat of the furnace.\* In sampling two conditions must be observed: First, the original sample should be of considerable size and thoroughly representative; and, second, the quartering down to an amount which can be put into a sealed "lightning" jar should be carried out as quickly as possible after the sample is taken. Careful samplings and careful treatment of samples are necessary to obtain reliable results, especially in the determination of moisture. The lumps are coarsely broken, and the whole spread out in a low circular heap. Diameters are drawn at right angles in it and opposite quarters taken, and treated similarly to the whole sample. The operation is continued until a sample of a few pounds is obtained. This is roughly crushed and samples taken at different points for the moisture determination; it is then further quartered down until a

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\* Report of Committee on Coal Analysis, J. Am. Chem. Soc., 21, 1116 *et seq.* (1899).



sample of 100 grams which passes a 60-mesh sieve is obtained.

The methods employed in the analysis of fuels are largely a matter of convention, various methods giving varied results; for example, it is well-nigh impossible to obtain accurately the percentage of moisture in coal, as when heated sufficiently hot to expel the water some of the hydrocarbons are volatilized.

**Moisture.**—Dry one gram of coal in an open crucible at  $104^{\circ}$ – $107^{\circ}$  C. for one hour. Cool in a desiccator and weigh covered. Where accuracy is required, determinations must also be made on the coarsely ground sample; this latter result is to be regarded as the true amount and corrections applied to all determinations where the powdered sample is used.\* †

**Volatile Combustible Matter and Coke.\***§—Place one gram of fresh, undried powdered coal in a platinum crucible having a tightly fitting cover. Heat over the full flame of a Bunsen burner for seven minutes by the watch. The crucible should be supported on a platinum triangle with the bottom six to eight centimeters above the top of the burner. The flame should be fully twenty centimeters high when burning free, and the determination should be made in a place free from drafts. The upper surface of the cover should burn clear, but the under surface should remain covered with carbon. To find “Volatile Combustible Matter” subtract the per cent of moisture from the loss found here. The residue in the crucible minus the ash represents the *Coke or Fixed Carbon*.

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\* Report of Committee on Coal Analysis, *loc. cit.*

† See also an article by Hale, Proc. Am. Soc. Mech. Eng. 1896.

§ Sommermeier, J. A. C. S. 28, 1002 (1906).

Certain non-coking coals suffer mechanical loss from the rapid heating.

**Carbon and Hydrogen.**—These are determined by burning the coal in a stream of air and finally in oxygen, the products of combustion, carbon dioxide and water, being absorbed in potassium hydrate and calcium chloride.

*Apparatus Required.*—Combustion-furnace similar to that shown in Fig. 15. Combustion-tube filled.

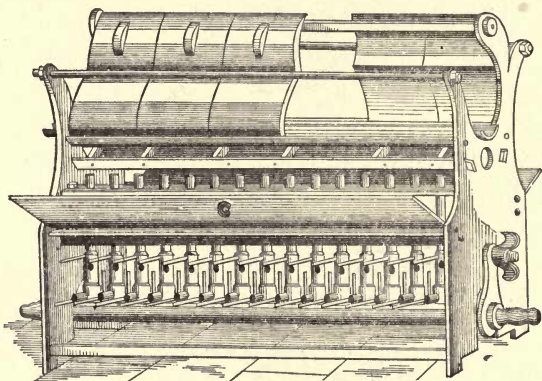


FIG. 15.—COMBUSTION-FURNACE.

Potash-bulbs with straight chloride of calcium tube filled. Chloride of calcium tube filled. Oxygen-holder, drying and purifying apparatus. Porcelain boat, desiccator, tongs,  $\frac{1}{8}$ -inch rubber tubing. Analytical balance.

The combustion-tube is of hard glass,  $\frac{1}{2}$  inch in internal diameter and 36 inches long, closed with perforated rubber stoppers. One end—called the front end—is filled with a layer of copper oxide 12 inches long, held in place by plugs of asbestos coming

within 4 inches of the stopper. In coals rich in sulphur the oxide is partially replaced by a layer of chromate of lead 2 inches long. The position of the boat containing the coal is immediately behind this copper oxide; behind the boat is placed an oxidized copper gauze roll, 6 inches long. Before making the combustion, the tube and contents should be heated to a dull red heat in a stream of oxygen freed from moisture and carbon dioxide by the purifying apparatus, to burn any dust and dry the contents; it is then ready for use.

The potash-bulbs are an aggregation of five bulbs, the three lowest filled with potassium hydrate of 1.27 sp. gr., the other two serving as safety-bulbs, preventing the liquid from being carried over into the connectors. They should be connected further with a chloride of calcium tube to absorb any moisture carried away by the dry gas. When not in use they should be closed with connectors carrying glass plugs. Before weighing they should stand at least fifteen minutes in the balance-room to attain its temperature; the weight should be to milligrams and without the connectors.

The chloride of calcium tube is of U form, provided with bulbs for the condensation of the water; the granular calcium chloride is kept in place by cotton plugs, and the stopper neatly sealed in with sealing-wax. As calcium chloride may contain oxide which would absorb the carbon dioxide formed, a current of dry carbon dioxide should be passed through the tube and thoroughly swept out by dry air before use.

The chloride of calcium tube like the potash-bulbs should be placed in the balance-room fifteen minutes

before weighing and, if the balance-case be dry, may be weighed without the connectors. It should be weighed to milligrams.

The oxygen-holder may be like the Muencke aspirator, Fig. 14. The oxygen should be purified by passing through potassium hydrate and over calcium chloride.

*Operation.*—The front stopper of the combustion-tube is slipped carefully upon the stem of the chloride of calcium tube and this connected to the potash-bulbs; 0.2 to 0.3 gram of the coal is carefully weighed into the porcelain boat (to 0.1 mg.), the roll removed, and the boat inserted behind the layer of copper oxide, and the roll and stopper replaced. The tube is now ready to be heated.

The front of the copper oxide is first heated, the heat being gradually extended back; at this time the rear end of the copper roll is heated and a slow current of purified air passed through. This method of gradual heating of the tube is followed until the layer of copper oxide and the rear portion of the roll are at a dull red heat. Heat is now cautiously applied to the coal and the current of air slackened. The volatile matter in the coal distils off, is carried into the layer of copper oxide and burned; the carbon dioxide formed can be seen to be absorbed by the potassium hydrate. When this absorption almost ceases, oxygen is turned on and the coal heated until it glows. The stream of oxygen should be so regulated as to produce but two bubbles of carbon dioxide in the bulbs per second. If the evolution be faster, the gas is not absorbed. When the coal has ceased glow-



ing, oxygen is allowed to pass through the apparatus until a spark held at the exit of the last chloride of calcium tube (on the bulbs) re-inflames; the oxygen is allowed to run for fifteen minutes longer. The current of oxygen is now replaced by purified air, and the heat moderated by turning down the burners and opening the fire-clay tiles; the air is allowed to run through for twenty minutes to thoroughly sweep out all traces of carbon dioxide and moisture. The bulbs and U tube are disconnected, stopped up, allowed to stand in the balance-room, and weighed as before. The increase in weight in the bulbs represents the carbon dioxide formed; this multiplied by the factor 0.2727 gives the carbon. Similarly the increase in the U tube, minus the water due to the moisture in the coal, represents the water formed, one ninth of which is hydrogen.

*Notes.*—At no time in the combustion should any water appear near the copper roll, as it is an indication that the products of combustion have gone backward into the purifying apparatus and hence are lost. Such analyses should be repeated. Should moisture appear in the front end, it may be *gently* heated to expel it. Both ends of the tube should be frequently touched with the hand during the combustion, and should be no hotter than may be comfortably borne, as the stoppers give off absorbable gases when highly heated. Care should be taken not to heat the tube too hot, fusing the copper oxide into and spoiling it. One tube should serve for a dozen determinations. It should not be placed upon the iron trough of the

furnace, but upon asbestos-paper in the trough, to prevent fusion to the latter.

As will be seen, the execution of a combustion is not easy, and should only be intrusted to an experienced chemist. The results obtained are usually 0.1 per cent too low for carbon and a similar amount too high for hydrogen.

**Ash.**—This is determined by weighing the residue left in the boat after combustion, or by completely burning one gram of the coal contained in a platinum dish; often a stream of oxygen is used.

**Nitrogen** is determined by Kjeldahl's method, which consists in digesting the coal with strong sulphuric acid, aided by potassium permanganate, until nearly colorless. The nitrogenous bodies are changed to ammonia, which forms ammonium sulphate and may be determined by rendering alkaline and distilling the solution.

**Sulphur** is determined by Eschka's method, consisting in heating for an hour one gram of the coal mixed with one gram of magnesium oxide and 0.5 gram. sodium carbonate in a platinum dish without stirring, using an alcohol-lamp, as gas contains sulphur. It is allowed to cool and rubbed up with one gram of ammonium nitrate and heated for 5 to 10 minutes longer. The resulting mass is dissolved in 200 cc. of water evaporated to 150 cc., acidified with hydrochloric acid, filtered, and sulphuric acid determined in the filtrate in the usual way with barium chloride.

**Oxygen** is determined by difference, there being no direct method known.

## ANALYSIS OF LIQUID FUELS.

**Carbon and Hydrogen.**—This determination is made as in the case of the solid fuels, the liquid being contained in a small bulb sealed for weighing to prevent volatilization. The stem is scratched and broken off and the bulb inserted in the combustion tube in place of the boat. Extra care in heating has to be observed to prevent the liquid from passing through unburnt. For thick or tarry oils having a small quantity of volatile matter, the boat may be used as with solid fuels.

**Sulphur.**—For oils containing more than 0.01 per cent sulphur the well known method of Carius may be employed. This consists in sealing up the oil contained in a small weighing tube, in a tube with fuming nitric acid and barium chloride and heating in a furnace for several hours. All sulphur is converted into sulphuric acid, which combines with the barium chloride forming barium sulphate, which is filtered off and weighed in the usual way. Another method consists in burning the oil in a small lamp and collecting the products of combustion. The lamp is a miniature "oil lamp" made from a 3-inch test-tube (weighing tube) by drawing a piece of weighed lamp wicking through a small glass tube contained in the stopper. This lamp is suspended by a wire from the balance and weighed accurately.

It is lighted and hung under a funnel arranged so that the products of combustion are drawn by an air-pump through a series of two washing bottles containing saturated bromine water. After about a gram of oil has been burned (about 1.3 cc.) the wick is carefully removed without losing any oil, the stopper replaced and the tube

again weighed. The wick and oil it contains are covered with Eschka's mixture and treated as for the determination of sulphur in coal (p. 78). The hydrochloric acid filtrate is added to the bromine solution, the bromine boiled out, the solution evaporated to about 150 cc., and the sulphuric acid formed determined in the usual way with barium chloride.

The oil burned is obviously the weight of the lamp before and after burning less the weight of the dry wick. This treatment of the wick is necessary, as Conradson\* has found sometimes 40 per cent of the sulphur in the wick.

Barlow's † method is excellent, but requires considerable experience to carry out.

**Nitrogen** is determined exactly as in the case of solid fuels.

**Water** can be shown qualitatively by the eosine test, ‡ by rubbing with a little eosine on a glass plate. If water be present the oil will take on a pink color. For its quantitative determination a weighed amount of gently ignited plaster of Paris is added to the oil and allowed to stand 24–36 hours. Gasoline is now added to the oil and the whole brought upon a dried weighed filter and the plaster washed until all oil is removed; the filter and contents are dried at a gentle heat not exceeding 100° C. to a constant weight. The increase in weight represents the quantity of water in the oil.

*Flash and Fire Test.*—Determined by heating the oil in the covered New York tester or lubricating oil tester according to Gill, "Short Handbook of Oil Analysis," Chapters I and II.

The analysis of gaseous fuels has already been described in Chapter V.

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\* J. Ind. and Eng. Chem., 2, 171 (1910).

† J. Am. Chem. Soc., 26, 341 (1904).

‡ Holley and Ladd, Mixed Paints, Color Pigments and Varnishes, p. 36.



DETERMINATION OF CALORIFIC POWER OF SOLID  
AND LIQUID FUEL.*a. Direct Methods.*

Many forms of apparatus have been proposed for this purpose; few, however, with the exception of those employing Berthelot's principle—of burning the substance under a high pressure of oxygen—have yielded satisfactory results. The apparatus of William Thomson,\* and also that of Barrus, in which the coal is burnt in a bell-jar of oxygen, while usually yielding results within 3 per cent of the calculated value, yet they may vary as much as 8 per cent from that value.† Unless a crucible lined with magnesia be used, or the sample mixed with bituminous coal, it is inapplicable to certain semi-bituminous and anthracite coals, as the ash formed over the surface prevents the combustion of the coal beneath it.

Fischer's calorimeter ‡ is similar in principle, but is claimed to give very good results.§

Lewis Thompson's calorimeter, in which the coal is burnt in a bell-jar by the aid of oxygen furnished by the decomposition of potassium chlorate or nitrate, is open to several objections, the chief of which are:  
1. The evolution of heat due to the decomposition of the

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\* Thomson, Jour. Soc. Chemical Industry, 5, 581.

† Ibid., 8, 525.

‡ Zeit. f. angewandte Chemie, 12, 351.

§ Bunte, Jour. f. Gasbeleuchtung und Wasserversorgung, 34, 21, 41.

oxidizing substance used. 2. Loss of heat due to moisture carried off by the gases in bubbling through the water. The results which it gives must be increased by 15 per cent.\*

Hempel's apparatus † makes use of the Berthelot principle: the coal must be compressed into a cylinder for combustion—a process to which every coal is not adapted—only applicable to certain varieties of bituminous and brown coal. The mixture with the coal of any cementing or inflammable substance to form these cylinders carries with it the necessity of accurately determining its calorific power beforehand.

The best apparatus for the purpose is probably that of Mahler ‡§ modified by Williams, Norton, and Emerson, the modifications consisting in replacing the enamel lining by a nickel one or by electroplating the inside with gold and in improved methods of making the apparatus tight.

**The Mahler apparatus**, Fig. 17, consists of a mild-steel cylinder *B*, with walls half an inch thick, narrowed at the top for connection by a screw-joint with the cover carrying the vessel *C* to contain the coal. This cylinder or bomb is placed inside the calorimeter *D*, and this inside a jacket *A*. At the right is shown a portion of the oxygen-cylinder and the gauge.

For the following directions for its use the author is indebted to the kindness of Professor Silas W. Holman of the Institute of Technology.

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\* Scheurer-Kestner, *J. Soc. Chem. Industry*, 7, 869 (1888).

† Mahler, *id.* 11, 840 (1892).

‡ Hempel, *Gasanalytische Methoden*, p. 347.

§ Mayer, *Stevens Indicator* (1895), 134.

\*\* For pressures developed in bomb, see Benedict and Fletcher, *J. Am. Chem. Soc.*, 29, 739 (1907).

**Preparation of Bomb.**—Remove the ring upon which it sits in the calorimeter.

Wash out the bomb. It need not be dry. Leave cover off.

See that the lead-ring washer *P*, Fig. 16, is in good condition. Unless its upper surface is fairly smooth the cover cannot be tightly closed. Repeated screwing on of the cover raises a burr of lead. When this becomes noticeable it must be removed by cutting with a knife-blade. If there is difficulty in making the cover tight, it is most likely to be due to this cause.

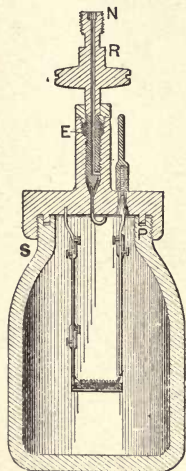


FIG. 16.—MAHLER'S BOMB.

Grease the screw *S* upon the outside of the bomb slightly with tallow or a heavy oil, but be sure that none of the grease gets beyond the lead washer.

Secure the bomb very firmly in the heavy clamp on the table.

Place the top on a ring or in a clamp of a lamp-stand and in an upright position.

Put in position the platinum tray *C* and the rod *E*, Fig. 17.

Twist on the loop of ignition-wire (fine platinum or iron). This must make good electrical contact with both *E* and the pan or its supporting rod. Failing this the current will not flow to fuse the wire. Failure to ignite is almost always traceable to this cause.

Pour into the tray a known weight of the substance

to be burned. If this be coal, slightly over one gram should be used. It is usually best inserted from a small test-tube weighed before and after, with due precautions against loss.

The ignition-wire should dip well into the coal.

The fineness required in the combustible depends

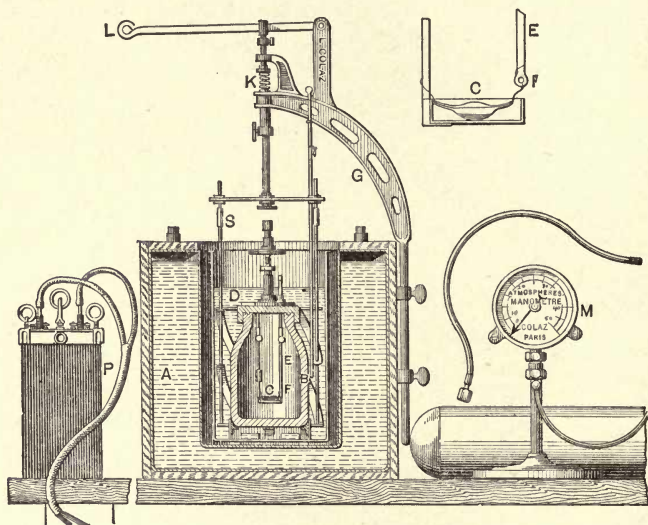


FIG. 17.—MAHLER'S APPARATUS COMPLETE.

upon its nature. Anthracite coal should be in a very fine powder, at least 100 mesh. Trial will show whether any unburned grains remain, indicating that the combustible is too coarse.

The standard which carries the pressure-gauge should be screwed to the table near the bomb-clamp, and the oxygen cylinder must be placed near by so that the three may be easily connected by the flexible copper tube.



The top carrying the charge is then cautiously (to avoid loss of charge by jarring or draft) transferred to the bomb and screwed carefully home. The lifting is best done by hooking the fingers beneath the milled head at the top of the valve-screw *R*. The top must be set up hard by the wrench which takes the large nut cut on the cover. In setting this up it is desirable to use no more force than is necessary to secure a gas-tight bearing of the tongue of the cover against the lead washer *P*. Just the force required can only be learned by experience, but it is always considerable. A slight leak is unimportant, but it is not difficult to secure a tight seal if the lead washer be kept in good condition.

To fill with oxygen proceed as follows:

Screw down the valve-screw *R* *gently* to close the valve. Connect the copper tube to the oxygen-tank gauge, and to the bomb at *N*. See that there are leather washers at the joints. Turn the connecting nuts firmly but not violently home. The connections to the oxygen-tank and gauge are usually left undisturbed, and only that at *N* has to be made each time.

It is now necessary to test for leakage in the connections. To do this, as *R* is closed, it is only necessary to open the oxygen-tank *cautiously* by means of its wrench until the gauge indicates 5 or 10 atmospheres and then close it. As the tank when freshly charged has a pressure of 120 atmospheres, and the gauge reads only to 35 atmospheres, *care must be used in all manipulations not to overstrain the gauge*, also avoid suddenly releasing the pressure on the gauge. When this pressure is on, any leak in the connections

will be indicated by a drop in the gauge reading. If a leak exists, it must be removed or rendered extremely slow before proceeding further. It is most likely to be found in the joints, which must be tightened one by one until the leak stops.

Now to fill the bomb it is next necessary to open *R*. This could be done by merely turning back the milled head, or the nut just above it. But as this would put a twist into the copper connecting-tube (which many times repeated would break it), the better way is, holding one wrench in each hand, to loosen the connecting nut above *N* by a half-turn, holding *R* by the wrench and nut, then to turn the nut open a half-turn or until it is again tight in. This leaves the connections tight and *R* open into the bomb. The oxygen is then turned slowly on, and the bomb gradually fills. If a gram of coal is to be burned, a pressure of 25 atmospheres gives the proper amount of gas in the bomb. Note that the valve *R* and the inlet-tube have small borings. Thus the inflow of gas will be slow and the pressure in the connecting-tube will be higher than in the bomb. If, therefore, the tank be closed quickly, the gauge-reading will fall somewhat until these pressures equalize, and will then remain stationary unless there is a leak. The tank-cock must always be kept well under control to avoid overcharging either gauge or bomb.

When the bomb is full, close first the tank-cock. Then, to close *R*, put the wrenches on the nuts and, holding one from turning, set the other down until *R* is tight, *but not too tight*. Avoid straining *R*, which closes tight *very easily*. By this method the copper

tube is not twisted. There is of course a slight leak of gas from the bomb after *N* leaves the nut and before *R* is closed, but the time required for the half-turn is so short and the outflow so slow that the loss is insignificant. There is *no need to hurry* in this operation. Be deliberate and careful of the apparatus. A valve like *R* is a nice piece of workmanship, and to endure much usage it must be treated with care.

The bomb is now ready to be unclamped and set into the ring preparatory to transfer to the calorimeter. It can be left standing indefinitely, but must be handled with caution (best by lifting with fingers beneath *R*, to avoid spilling the charge).

**Preparation of Calorimeter.**—The outer jacket of the calorimeter should be filled with water at about the room temperature or a few degrees higher. If left standing from day to day it will usually be nearly enough right. It is well to stir it (blow air through it) somewhat before beginning work, if it has stood for some time.

Be sure that the inner surface of this jacket, i.e., the one which is next the calorimeter, is *thoroughly dry*, and do not let any water spill into it—or remove it if it does so.

*Thoroughly dry* the outer surface of the calorimeter and *keep it so*. Moisture depositing on or evaporating from the surface of the calorimeter is sure to cause an irregular error which may spoil otherwise good work.

Put the calorimeter in place. Transfer the bomb to it, and adjust the stirrer so that it works properly.

Pour in the proper amount of water, about 2.25

liters, at a suitable temperature, best by using marked flasks carefully calibrated beforehand.

Insert the thermometer.

See that the electrical attachments are ready for instantaneous use. The whole is then ready for the combustion.

**Combustion Observations.**—With apparatus all in place run the stirrer briskly and continuously until the completion of the work. Allow about five minutes for everything to come to a normal condition. Then take temperature readings to at least  $0.01^{\circ}$  at each quarter minute for at least five minutes. Record the times (h. m. s.) and corresponding thermometer-readings, thus:

Time.	Temp.	Remarks.
2 <sup>h</sup> 15 <sup>m</sup> 0 <sup>s</sup>	15°.24	After 5 <sup>m</sup> stirring
15	.24	
30	.25	
45	.25	
16 0	.25	
15	.26	
30	.26	
. . . . .		
25 0	—	Coal ignited
15	15.6	
30	.9	
45	16.2	
26 0	.5	
etc.	etc.	

Exactly at the beginning of a noted minute close the electric circuit through the fuse-wire. If the arrangements are right, this will cause the coal to ignite at once and the combustion is almost instantaneous. Owing to the time required to transmit the



heat through the bomb to the water, the temperature, however, will continue to rise for two or three minutes. Keep up the steady stirring and the quarter-minute temperature-readings for at least ten minutes after ignition, recording as above. One or two observations may be unavoidably lost before and after ignition, but this does not materially affect the results. The readings during the rapid rise are also less close.

As soon as the rise begins to slow down, however, the hundredths of a degree must again be secured. This makes a series of observations of 15 to 20 minutes' duration. The use of the readings to obtain the cooling correction and the corrected rise of temperature of the calorimeter is given under the heading "Cooling Correction" farther on.

This completes the observations unless it is desired to test the character of the products of combustion. The bomb should now be opened and rinsed, as the nitric acid formed by the oxidation of the nitrogen in the coal and air attacks the metallic lining unless it be of gold. Also the top is more easily unscrewed at first than later. Leave the top off.

*Before unscrewing the top of the bomb be sure to open the valve R to relieve the pressure.*

**Heat Capacity of Bomb and Calorimeter.**—The heat capacity of the bomb may be found:

1. From the weights and assumed specific heats of the parts.
2. By raising the bomb to an observed high temperature and immersing in water, i.e., by the usual "method of mixtures."
3. By burning in it a substance of known heat of

combustion, such as pure naphthaline, and calculating back to find the heat capacity of the bomb.

The first method is not reliable. Errors of several per cent may enter in the assumed specific heats.

The second method is very difficult of exact performance, owing to the size and form of the bomb.

The third method is by far the most reliable, but of course depends on the correctness of the assumed heat of combustion of the substance used. That of naphthaline has been so well determined by Berthelot and others,\* and the substance is so easily and cheaply obtained in a pure state, that dependence can be placed on the results. This method has the great advantage that it involves the use of the apparatus in precisely the same way as in subsequent determination, so that any systematic errors of method tend to cancel one another. It also determines at the same time the heat capacity of the calorimeter and stirrer just as used.

The capacity of the calorimeter and stirrer may best be determined in connection with that of the bomb by the third method just described. Otherwise it may be found by the first method, or by a method similar to the second, viz., by pouring into the calorimeter when partly full water of a known temperature different from that of the water in the calorimeter, noting all temperatures and weights. This last method, however, is very unsatisfactory in practice owing to the small heat capacity of the calorimeter and to the losses of heat in pouring the water, etc.

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\* 1 gram of naphthaline evolves 9692 C. This is the average of 150 determinations by four different observers.

A general expression for computing the heat of combustion from the bomb observations is as follows: Let  $n$  represent the number of grams of combustible,  $H$  the heat of combustion sought,  $W$  the weight of water in the calorimeter, and  $k$  the heat capacity, or water equivalent, of bomb, calorimeter, stirrer, thermometer, etc.;  $t_1$  and  $t_2$  represent the initial and final temperatures of the water. Then

$$nH = W(t_2 - t_1) + k(t_2 - t_1),$$

whence

$$H = \frac{1}{n}(W + k)(t_2 - t_1).$$

This expression is exact if  $t_2$  is corrected for loss by cooling as described in the methods for "Cooling Correction," p. 85.

The value of  $k$  may be determined by either of the following methods; a simplification may, however, be introduced which will save much labor if an accuracy of not more than about one per cent is sought, provided that  $k$  is found by burning naphthaline or other known substance. Use enough of this substance to cause about the same rise,  $t_2 - t_1$ , (within  $1^\circ$ ) as will be caused by one gram of coal. Omit the cooling correction entirely, using for  $t_2$  the maximum temperature attained. Then compute  $k$ ; this value will be erroneous by a small amount owing to the neglect of the correction. Now in subsequent measurements on coal also neglect the cooling correction, using for  $t_2$  the maximum observed temperature as before, thus leaving an error in  $t_2$ . Since the rise  $t_2 - t_1$  in both cases will be nearly the same, the error in  $k$  will almost

exactly affect that in  $t_2$  in the coal-test, and the resulting value of  $H$  will be nearly free from this error. This method of course implies that  $W$  is nearly constant and that  $t_1$  is systematically arranged to be either about at the air-temperature or a definite amount below it, as described under "Cooling Correction," so that the cooling loss is about the same. The time-interval from  $t_1$  to  $t_2$  must for the same reason be nearly constant in all cases.

**Cooling Correction.**—In all careful calorimetric work, one of the most troublesome sources of error is the loss or gain of heat by the calorimeter from its surroundings. This loss or gain is due to radiation, to air-convection currents, and to evaporation or condensation. Unavoidable irregularities in the conditions and the smallness of the quantities to be measured render the amount of the correction variable and its determination uncertain. Many methods of making the correction have been proposed. One of the best of these is the first of the two given below, but the second, although a little more troublesome in the execution of the work, appears to be more trustworthy in its results. The second method is to be used.

*First Method.*—This is described in the Physical Laboratory Notes, I,\* under "Specific Heat of Solids." In this method the water at the outset should be at such a temperature that it is gaining very slowly. For an open calorimeter this is about  $1^\circ$  or  $2^\circ$  below the air-temperature, but varies with circumstances. Water which has been long standing in the room is generally about right.

*Second Method.*—For the discussion and details

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\* Obtainable from A. D. Machlachlan, Bookseller, Boston.



reference may be had to an article by Professor Holman in Proc. American Academy of Arts and Sciences, 1895, p. 245; also in The Technology Quarterly, 8, 344.

**Parr's Calorimeter.\***—This “has the advantage of operating without an oxygen gas supply; its manipulation is simple and the extraction of the heat rapid, owing to the compact mass in which the heat is generated. It is especially adapted to soft coal, and while designed for technical purposes, its factor of error is well within 0.5 per cent.” “It depends for its action upon the liberation of oxygen from a compound which shall in turn absorb the products of combustion,

conditions admirably met in sodium peroxide; this obviates the necessity of providing for an outlet for those gases and also any loss arising from the heat they might carry off.”

“A, Fig. 18, is the calorimeter of about two liters capacity, insulated by two outer vessels of indurated fiber, B and C, so placed as to provide further insulation by the air-spaces b and c.

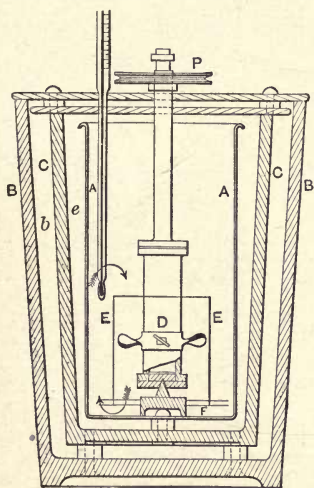


FIG. 18.—PARR'S CALORIMETER. The cover is double, to correspond, with an air-space between, the two parts being connected for convenience in handling. The cartridge *D* has a capacity of about 25 cc.; it rests on a pivot below, extends through the covers, and has a small removable pulley at the end. Turbine wings

\* Parr, J. Am. Chem. Soc., 22, 646 (1900).

fastened to spring clips are placed on the cartridge, and a short cylinder *E*, open at both ends, is provided for directing the current set up by rotation of the vanes attached to the cartridge. The stem *G* of the cartridge is so arranged as to permit the passage of a short piece of No. 12 copper wire to ignite the charge; it is provided with a valve *D* at the lower end to prevent the escape of the enclosed air."

**Manipulation.**—One gram of coal ground to pass a 100-mesh sieve, dried at  $105^{\circ}$ , is put into the cartridge, 16–18 grams of sodium peroxide added, the top screwed on, and the whole shaken to thoroughly mix the contents. The peroxide should be fine enough to pass a 25-mesh sieve. The cartridge is tapped to settle the charge to the bottom, placed in the calorimeter, two liters of water poured in, and rotated 50 to 100 revolutions per minute. The water should be 3 to 4 degrees lower than the room temperature. When the temperature has become constant, the thermometer is read, a hot wire dropped down *G*, igniting the charge, which burns completely. The extraction of the heat is effected in about five minutes; the reading of the maximum temperature is taken and the calculations made as follows: The rise of temperature is corrected, first, for that produced by the hot wire; this amounts to  $0.006^{\circ}$  C. per  $\frac{1}{4}$  inch of No. 12 copper wire: second, for the heat produced by the combination of the sodium peroxide with the carbon dioxide and water formed by the combustion; this amounts to 27 per cent of the total indicated heat. If  $C$  = the heat of combustion of the coal,  $C'$  the calories indicated,  $t$  the rise of temperature, and  $w$  the water employed, then

$$C' = (t - 0.006^\circ) \times w, \quad C = C' - \frac{C' \times 27}{100},$$

$$C = (t - 0.006^\circ) \times w \times 0.73.$$

*Notes.*—Instead of using a gram of coal some prefer to use half this quantity, mixing it thoroughly and rapidly with the peroxide in a watch-glass with a spatula and transferring it to the combustion-chamber.

Ignition by an electrically heated platinum wire is to be preferred to that by dropping a hot copper wire into the mixture. Accidents have been caused from the failure of the valve to work.

The combustion-chamber should be perfectly dry within and without.

Ashes and coke are difficult of ignition: this can be effected by adding a second charge of peroxide with half a gram of good coal, the combustion factor of which has been determined, and thoroughly mixing the charges and repeating the ignition.

For stirring, the smallest size electric or water motor furnishes sufficient power.

In the formulæ  $w$  = the water employed + the water equivalent of the calorimeter.

*Berthier's Method.* — Another method of direct determination was proposed by Berthier in 1835.\* It uses as a measure of the heating value the amount of lead which a fuel would reduce from the oxide; in other words, it is proportional to the amount of oxygen absorbed.

The method is as follows †: Mix one gram of the

\* Dingler's Polytechnisches Journal, 58, 391.

† Noyes, McTaggart & Craver, J. Am. Chem. Soc., 17, 847 (1895).

finely powdered dry coal with 60 grams of oxide of lead (litharge) and 10 grams of ground glass. This mixing can be done with a palette-knife on a sheet of glazed paper; the mixture is transferred to a fire-clay crucible (Battersea C size), covered with salt, the crucible covered and heated to redness in a hot gas-furnace—or the hottest part of the boiler-furnace—for 15–20 minutes. After cooling, the crucible is broken and the lead button carefully cleaned and weighed. Multiply the weight of the lead button obtained by 268.3 calories (or 483 B. T. U.) and divide the product by the weight of coal taken. The result is the number of calories per gram or B. T. U. per pound. One gram of lead is theoretically equivalent to 234 calories (C); owing to the hydrogen present this factor gives results about two per cent too low. The results obtained by the author using “horn-pan” scales in one case by this method were within 2.8 per cent of those yielded by the bomb calorimeter, which are as close as those obtained by any calorimeter save Parr’s. The method would seem worthy of more attention than it has received.

*b. Determination of Heating Value by Calculation.*

The method of determination of the heating value first described, though exact, has the disadvantages that the apparatus is costly and the compressed oxygen is not easily obtained. To obviate these, it has been sought to obtain the heating value by calculation from the chemical analysis, the heating value of the constituents being known. This has the disadvantage that we have no absolute knowl-



edge—nay, not even an approximate idea—as to how the carbon, hydrogen, water, and sulphur exist in the coal, so that any formula must of necessity be quite removed from the truth. Dulong was the first to propose the method by calculation, and his formula \* is

$$H = \frac{8000c + 34500(h - \frac{1}{8}o)}{100},$$

$c$ ,  $h$ , and  $o$  representing the percentages of carbon, hydrogen, and oxygen in the coal.

Many modifications of this, considering the water formed, the heat of vaporization of carbon, or the volatile hydrocarbons, have been proposed.

Bunte † finds that the following formula ‡ gives results varying from + 2.8 to - 3.7 per cent:

$$\frac{8080c + 28800\left(h - \frac{o}{8}\right) + 2500s - 600w}{100}.$$

$s$  and  $w$  represent the percentages of sulphur and water respectively: It is, however, inapplicable to anthracite coal. It would scarcely seem that the sulphur would be worth considering unless high, one per cent affecting the result but 0.3 per cent.

Mahler employs the formula \*

$$\frac{8140c + 34500h - 3000(o + n)}{100},$$

$o$  and  $n$  representing oxygen and nitrogen, and states that it gives results within 3 per cent.

The results obtained by these formulæ for anthracite coal are as a rule considerably too low.

\* H burned to liquid water. † H burned to aqueous vapor.

† Jour. für Gasbeleuchtung, 34, 21-26 und 41-47.

Goutal \* has proposed the formula

$$P = \frac{8150c + AM}{100}$$

as being more readily applicable than the preceding. This gives the results in calories.

$P$  represents the calorific power, expressed in calories;  $c$ , the percentage of fixed carbon (coke—ash);  $M$ , the percentage of volatile matter ( $100 - [\text{coke} + \text{ash} + \text{water}]$ );  $A$  is a coefficient which varies with the amount of volatile matter  $M$ , viz.,

$M = 2$ to $15$	$A = 13000$
15 to 30	10000
30 to 35	9500
35 to 40	9000

The results upon a series of American coals varied less than 2 per cent from those obtained by the calorimeter. It is, however, only applicable to high-grade coals,

REFERENCES.—An admirable discussion of the errors incident to the use of the Mahler calorimeter and others of that type will be found in a paper by G. A. Fries, Bulletin No. 94 (1907), U. S. Dept. of Agriculture, Bureau of Animal Industry, "Investigations in the Use of the Bomb Calorimeter."

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\* Revue de chimie ind., 7 (1896), 65; abs. in The Analyst, 24, 107.

## CALORIFIC POWER OF GASEOUS FUEL.

*a. Direct Determination.*

Perhaps the best apparatus for the determination of the heating value of gases is the Junkers calorimeter, Figs. 19 and 20. The following description is taken

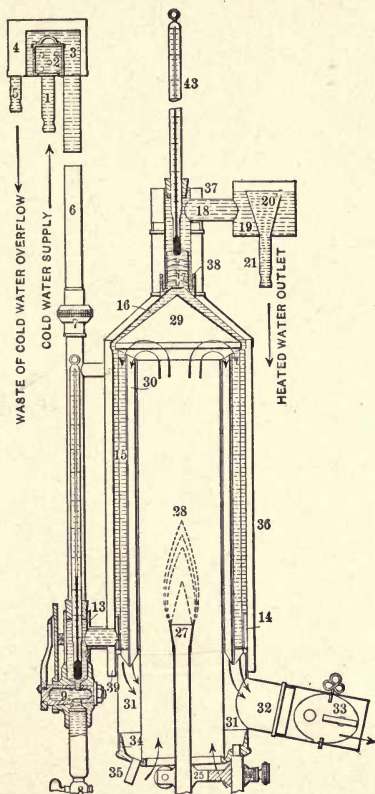


FIG. 19.—JUNKERS GAS-CALORIMETER (SECTION).

from an article by Kühne in the Journal of the Society of Chemical Industry, vol. 14, p. 631. As will be

seen from Fig. 19, this consists of a combustion-chamber, 28, surrounded by a water-jacket, 15 and 16, this being traversed by a great many tubes. To prevent loss by radiation this water-jacket is sur-

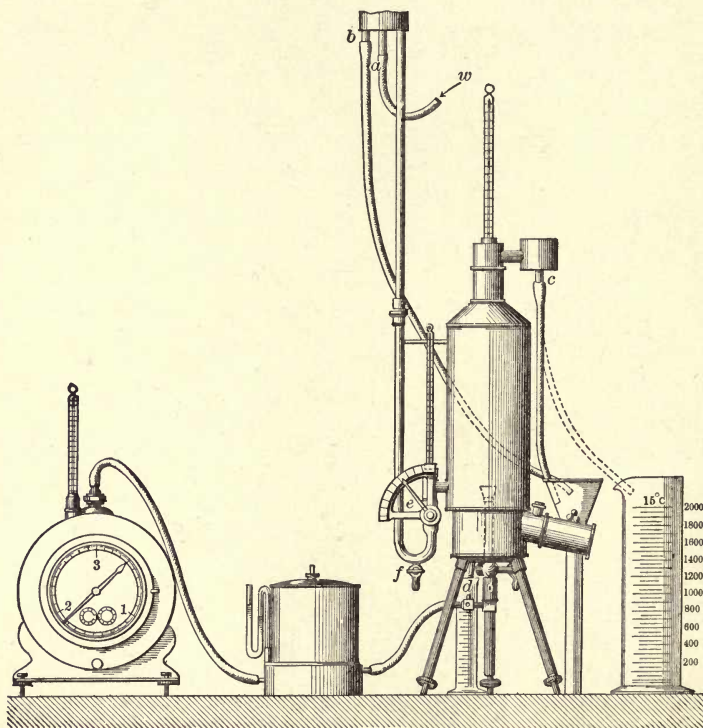


FIG. 20.—JUNKERS GAS-CALORIMETER.

rounded by a closed annular air-space, 13, in which the air cannot circulate. The whole apparatus is constructed of copper as thin as is compatible with strength. The water enters the jacket at 1, passes down through 3, 6, and 7, and leaves it at 21, while



the hot combustion-gases enter at 30 and pass down, leaving at 31. There is therefore not only a very large surface of thin copper between the gases and the water, but the two move in opposite directions, during which process all the heat generated by the flame is transferred to the water, and the waste gases leave the apparatus approximately at atmospheric temperature. The gas to be burned is first passed through a meter, Fig. 20, and then, to insure constant pressure, through a pressure-regulator. The source of heat in relation to the unit of heat 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 water and overflow constant. The temperatures of the water entering and leaving the apparatus can be read by 12 and 43; as shown before, the quantities of heat and water passed through the apparatus are constant. As soon as the flame is lighted, 43 will rise to a certain point and will remain nearly constant.

**Manipulation.**—The calorimeter is placed as shown in Fig. 20, 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 draft of air must be permitted to strike the exhaust of the spent gas.

The water-supply tube *w* is connected with the nipple *a* in the centre of the upper container; the other nipple, *b*, is provided with a waste-tube to carry away the overflow, which latter must be kept running while the readings are taken.

The nipple *c* through which the heated water leaves the calorimeter is connected by a rubber tube with the large graduate. *d* empties the condensed water into the small graduate.

The thermometers being held in position by rubber stoppers and the water turned on by *e* until it discharges at *c*, no water must issue from *d* or from 39, Fig. 19, as this would indicate a leak in the calorimeter.

The cock *e* is now set to allow about two liters of water to pass in a minute and a half, and the gas issuing from the burner ignited. Sufficient time is allowed until the temperature of the inlet-water becomes constant and the outlet approximately so; the temperature of the inlet-water is noted, the reading of the gas-meter taken, and at this same time the outlet-tube changed from the funnel to the graduate. Ten successive readings of the outflowing water are taken while the graduate (2-liter) is being filled and the gas shut off.

A better procedure is to allow the water to run into tared 8-liter bottles, three being used for a test, and weighing the water. The thermometer in the outlet can then be read every half-minute.

EXAMPLE.—Temp. of incoming water, 17.2°

“ “ outgoing “ 43.8°

Increase, 26.6°

Gas burned, 0.35 cu. ft.

$$\text{Heat} = \frac{\text{liters water} \times \text{increase of temp.}}{\text{cu. ft. gas}} = \frac{2 \times 26.6}{0.35} = 152.3\text{C.}$$

From burning one cubic foot of gas 27.25 cc. of water were condensed. This gives off on an average 0.6 C. per cc.

$27.25 \times 0.6 = 16.3$  C.;  $152.3 - 16.3 = 136$  C. per cubic foot;  $136 \times 3.96823 = 540$  B. T. U.

The calorific power obtained without subtracting the heat given off by the condensation of the water represents the *total* heating value of the gas. This is the heat given off when the gas is used for heating water or in any operation where the products of combustion pass off below  $100^{\circ}$  C. The *net heating value* represents the conditions in which by far the greater quantity of gas is consumed, for cooking, heating and gas engines, and is the one which should be reported. It should, however, be corrected,\* as shown on page 106, to the legal cubic foot, that is, measured at 30 inches barometric pressure, and  $60^{\circ}$  F. saturated with moisture.

The apparatus has been tested for three months in the German Physical Technical Institute with hydrogen, with but a deviation of 0.3 per cent from Thomson's value. This value may vary nearly that amount from the real value owing to the method which he employed.

### *b. By Calculation.*

Oftentimes it may be impracticable to determine the heating value of gases directly; in such cases recourse must be had to the calculation of its calorific power from volumetric analysis of the gas.

To this end multiply the percentage of each constituent by its number as given in Table IV, and the sum of the products will represent the British Thermal Units evolved by the combustion of one cubic foot of

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\* A difference of  $1^{\circ}$  C. or of 3 mm. pressure makes a change of 0.3 per cent in the volume. Pfeiffe, J. Gasbeleucht. 50, 67 (1907).

the gas.\* It is assumed that the temperature of the gas burned and the air for combustion is 60° F., and that of the escaping gases is 328° F., that corresponding to the temperature of steam at 100 pounds absolute pressure.

As has been already stated, column 3 in Table IV is based upon the assumption that the gas, and air for its combustion, enter at 60° F., and the products of combustion leave at 328° F.; in column 4 it is assumed that the entering temperature of both gas and air is 32° F., and the combustion-gases are cooled to 32° F. In case these conditions are varied, the amount of heat which the gas and air bring in must be determined; this is found in the usual way by multiplying the proportionate parts of 1 cubic foot, as shown by the analysis, by the specific heat of the gas, and this by the rise in temperature (difference between observed temperature and 32° F.). The quantity of air necessary for combustion is found by multiplying the percentage composition of the gas by the number of cubic feet necessary for the combustion of each constituent.

An example will serve to make this clear. The analysis of Boston gas is as follows:\*

CO <sub>2</sub>	"Illuminants."	O	CO	CH <sub>4</sub>	H	N
2.9	15.0	0.0	25.3	25.9	27.9	3.0

Or in one cubic foot there are

---

\* H. L. Payne, *Jour. Analytical and Applied Chem.*, 7, 230.

† Jenkins, *Annual Report Inspector of Gas Meters and Illuminating Gas*, 1896, p. 11.



.029 CO <sub>2</sub> .....	.259 CH <sub>4</sub>
.150 "illuminants".....	.279 H
.253 CO.....	.030 N

Let us assume that the gases, instead of passing out at a temperature of 328° F., leave at the same temperature as that of the chimney-gases, p. 29, 250° C. or 482° F.

The calculation of the heat carried away is similar to that there given.

0.15 cu. ft. of "illuminants" produces, Table III, 0.3 cu. ft. CO<sub>2</sub> and 0.3 cu. ft. steam;

0.253 cu. ft. of carbonic oxide produces .253 cu. ft. CO<sub>2</sub>;

0.259 cu. ft. methane produces 0.259 cu. ft. CO<sub>2</sub> and .518 cu ft. steam;

0.279 cu. ft. hydrogen produces .279 cu. ft. steam.

From the combustion of the gas there results .812 cu. ft. CO<sub>2</sub>, 1.097 cu. ft. steam, and 5.90 × 79.08 or 4.665 cu. ft. N.

The quantity of heat they carry off is as follows:

	Vol.	Vol. Sp. Ht.	Rise.	B.T.U.
CO <sub>2</sub> .....	.812 ×	.027 ×	450 =	9.9
N.....	4.66 ×	.019 ×	450 =	39.9
Excess of air..	1.2 ×	.019 ×	450 =	10.2
Steam.....	1.097 ×	.0502 ×	1229 =	67.7
Total heat lost..... =				127.7

The loss due to the steam is found by multiplying the weight of steam found by the "Total Heat of Steam," as found from Steam Tables.\* The tables, however, do not extend beyond 428° F.; it can be calculated by the formula

$$\text{Total heat} = \lambda = 1091.7 + 0.305(t - 32).$$

One cubic foot of hydrogen when burned yields .0502 lbs. of water.

The heat generated by the combustion of the gas is found by multiplying its volume by its calorific power, Table IV.

"Illuminants" . . . .	0.15	×	2000.0	=	300.0	B.T.U.	
CO . . . . .	0.253	×	341.2	=	86.3		
CH <sub>4</sub> . . . . .	0.259	×	1065.4	=	276.0		
H . . . . .	0.279	×	345.4	=	96.3		
						<u>        </u>	
Heat generated by the gas. . . . .					758.6	B.T.U.	
						<u>        </u>	
Total heat lost (p. 98) . . . . .					127.7		
						<u>        </u>	
						630.9	B.T.U.

This figure, 630.9 B.T.U., represents the heating power of one cubic foot of the gas measured at 62° F., and is consequently too small; its heating value at 32° F. is represented by

$$\frac{492 + 30}{492} \times 630.9, \text{ or } 669.1 \text{ B.T.U.}$$

The above calculation, like all giving accurate results, is somewhat tedious; a shorter and less correct one is as follows: Divide the figures found in the last column of

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\* Peabody's Steam Tables.

Table IV of the Appendix by 100, the result gives the heating value of these gases in B.T.U. per cubic centimeter.\* According to the volumetric analysis of the gas there are in 100 cc. the following:

15.0 cc. illuminants,	25.3 cc. carbonic oxide;
29.5 cc. methane,	27.9 cc. hydrogen;

the heating value is

$$15.0 \times 20.0 = 300.0 \text{ B.T.U.}$$

$$25.3 \times 3.41 = 86.3$$

$$25.9 \times 10.65 = 276.0$$

$$27.9 \times 3.45 = 96.3$$

$$\underline{\hspace{1.5cm}} \\ 758.6 \text{ B.T.U.}$$

the same as the gross heating value obtained by the other method. No correction is applied for the heat lost.

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\* Method followed in Prof. Paper, No. 48, U. S. Geol. Survey, Part III, p. 1005.





## APPENDIX.

### TABLE I.

TABLE SHOWING THE TENSION OF AQUEOUS VAPOR AND ALSO THE WEIGHT IN GRAMS CONTAINED IN A CUBIC METER OF AIR WHEN SATURATED.

From 5° to 30° C.

Temp.	Tension, mm.	Grams.	Temp.	Tension, mm.	Grams.	Temp.	Tension, mm.	Grams.
5	6.5	6.8	14	11.9	12.0	23	20.9	20.4
6	7.0	7.3	15	12.7	12.8	24	22.2	21.5
7	7.5	7.7	16	13.5	13.6	25	23.6	22.9
8	8.0	8.1	17	14.4	14.5	26	25.0	24.2
9	8.5	8.8	18	15.4	15.1	27	26.5	25.6
10	9.1	9.4	19	16.3	16.2	28	28.1	27.0
11	9.8	10.0	20	17.4	17.2	29	29.8	28.6
12	10.4	10.6	21	18.5	18.2	30	31.5	29.2
13	11.1	11.3	22	19.7	19.3			

### TABLE II.

“VOLUMETRIC” SPECIFIC HEATS OF GASES.\*

Air.....	0.019	“Illuminants”.....	0.040
Carbon dioxide.....	0.027	Methane.....	0.027
Carbonic oxide.....	0.019	Nitrogen.....	0.019
Hydrogen.....	0.019	Oxygen.....	0.019

The “volumetric” specific heat is the quantity of heat necessary to raise the temperature of one cubic foot of gas from 32° F. to 33° F.

\* H. L. Payne, *Jour. Anal. and Applied Chem.*, 7, 233.

TABLE III.

THE VOLUME OF OXYGEN AND AIR NECESSARY TO BURN ONE CUBIC FOOT OF CERTAIN GASES, TOGETHER WITH THE VOLUME OF THE PRODUCTS OF COMBUSTION.

Name.	Formula.	Volume of Oxygen.	Volume* of Air.	Volume of Steam.	Volume of Carbon Dioxide.	Ignition Point Deg F.
Hydrogen . . . .	H <sub>2</sub>	0.5	2.39	1	0	1085
Carbonic oxide	CO	0.5	2.39	0	1	1200
Methane . . . . .	CH <sub>4</sub>	2.0	9.56	2	1	1230
Ethane . . . . .	C <sub>2</sub> H <sub>6</sub>	3.5	16.73	3	2	1140
Propane . . . . .	C <sub>3</sub> H <sub>8</sub>	5.0	23.90	4	3	1015
Butane . . . . .	C <sub>4</sub> H <sub>10</sub>	6.5	31.07	5	4	....
Pentane . . . . .	C <sub>5</sub> H <sub>12</sub>	8.0	38.24	6	5	....
Hexane . . . . .	C <sub>6</sub> H <sub>14</sub>	9.5	45.41	7	6	1400
Ethylene † . . . .	C <sub>2</sub> H <sub>4</sub>	3.0	14.34	2	2	1010
Propylene † . . . .	C <sub>3</sub> H <sub>6</sub>	4.5	21.51	3	3	940
Benzene § . . . . .	C <sub>6</sub> H <sub>6</sub>	7.5	35.85	3	6	....
Acetylene . . . . .	C <sub>2</sub> H <sub>2</sub>	2.5	11.95	1	2	788

\* Air being 20.92 per cent by volume, 4.78 volumes contain 1 volume of oxygen.

† The chief constituent of "illuminants," new name "ethene."

‡ New name "propene."

§ Often called benzol, not to be confounded with benzine.

|| Dixon & Coward, Proc. Chem. Soc., 26, 67.

TABLE IV.

CALORIFIC POWER OF VARIOUS GASES\* IN BRITISH THERMAL UNITS PER CUBIC FOOT.

Name.	Symbol.	60° initial. 32° final. §	32° initial. 32° final.
Hydrogen.....	H	263.2	345.4
Carbonic oxide.....	CO	300.9	341.2
Methane.....	CH <sub>4</sub>	853.0	1065.0
Illuminants †.....	....	1700.0	2000.0
Ethane.....	C <sub>2</sub> H <sub>6</sub>	.....	1861.0
Propane.....	C <sub>3</sub> H <sub>8</sub>	.....	2657.0
Butane.....	C <sub>4</sub> H <sub>10</sub>	.....	3441.0
Pentane.....	C <sub>5</sub> H <sub>12</sub>	.....	4255.0
Hexane †.....	C <sub>6</sub> H <sub>14</sub>	.....	5017.0
Ethylene.....	C <sub>2</sub> H <sub>4</sub>	.....	1674.0
Propylene.....	C <sub>3</sub> H <sub>6</sub>	.....	2509.0
Benzene.....	C <sub>6</sub> H <sub>6</sub>	.....	4012.0
Acetylene.....	C <sub>2</sub> H <sub>2</sub>	.....	1477.0

\* H. L. Payne, *loc. cit.*

† Where the "illuminants" are derived chiefly from the decomposition of mineral oil.

‡ The chief constituent of the "gasolene" used in the gas machines for carburetting air.

§ The temperature of steam at 100 lbs. absolute pressure.

TABLE V.

CALORIFIC POWER OF VARIOUS LIQUIDS IN BRITISH THERMAL UNITS PER POUND.

Name.	B. T. U.	Pounds of Air for Combustion.
Crude petroleum, Lima, Ohio.....	20,890	
Crude petroleum, Oil Creek, Pa.....	21,600	
Crude petroleum, heavy, W. Va.....	18,180	
63° Bé. gasolene.....	19,162-20,448	14.96*
114° F. flash kerosene.....	18,643-19,892	14.89*
150° fire-test kerosene.....	18,290	
Mineral sperm.....	20,065	
Ethyl alcohol, 95 per cent.....	10,504	
Methyl alcohol.....	9,565	
Denatured alcohol.....	10,512-11,616	8.13*

\* Bull. 43 Bureau of Mines, 1912, pp. 18-21. "Fuel Values of Gasolene and Denatured Alcohol in Internal Combustion Engines."

TABLE VI.

CALORIFIC POWER OF CERTAIN SOLID FUELS IN BRITISH THERMAL UNITS  
PER POUND, FIGURED ON THE PURE DRY COMBUSTIBLE.

Anthracite.....	13,740-15,620
Bituminous coal:	
Cumberland.....	16,320
Georges Creek.....	15,140
Pocahontas.....	15,700
West Virginia.....	13,700
Brown coal.....	9,060-14,240
Coke.....	13,880-14,560
Peat.....	7,400-10,625
Tanbark.....	6,100
Wheat straw.....	10,380
Wood, hard.....	8,500
Wood, pine.....	9,150

TABLE VII.

SHOWING THE WEIGHT OF A LITER AND SPECIFIC GRAVITY REFERRED  
TO AIR, OF CERTAIN GASES AT 0° C. AND 760 MM.

Name of Gas.	Weight, Grams.	Specific Gravity.
Carbonic oxide.....	1.251	0.967
Carbon dioxide.....	1.966	1.519
Hydrogen.....	0.0896	0.069
Methane.....	0.715	0.553
Nitrogen.....	1.255	0.970
Oxygen.....	1.430	1.105
Air.....	1.294	1.000



TABLE VIII.

## SOLUBILITY OF VARIOUS GASES IN WATER.

One volume of water at 20° C. absorbs the following volumes of gas reduced to 0° C. and 760 mm. pressure.

Name of Gas.	Symbol.	Volumes.
Carbonic oxide.....	CO	0.023
Carbon dioxide.....	CO <sub>2</sub>	0.901
Hydrogen.....	H <sub>2</sub>	0.019
Methane.....	CH <sub>4</sub>	0.035
Nitrogen.....	N <sub>2</sub>	0.014
Oxygen.....	O <sub>2</sub>	0.028
Air.....	....	0.017

TABLE IX.

## MELTING-POINTS OF VARIOUS METALS AND SALTS, FOR USE WITH APPARATUS FIG. II.

Alphabetically.		By Temperatures.	
Aluminium.....	658° C.	Tin.....	232° C.*
Antimony.....	630	Bismuth.....	270*
†Barium chloride.....	950	Cadmium.....	302*
Bismuth.....	270	Lead.....	327*
Calcium fluoride.....	902	Zinc.....	419*
Cadmium.....	302	Cadmium chloride.....	541‡
†Cadmium chloride.....	541	Antimony.....	630*
Copper.....	1083	Aluminium.....	658*
Lead.....	327	Potassium bromide.....	740*
†Potassium bromide....	740	Sodium bromide.....	748*
†Potassium chloride....	780	Potassium chloride.....	780*
†Sodium bromide.....	748	Sodium carbonate.....	853*
†Sodium carbonate.....	853	Calcium fluoride.....	902§
Tin.....	232	Barium chloride.....	950*
Zinc.....	419	Copper.....	1083*

\* Burgess-Le Chatelier, Measurement of High Temperatures, 1912.

† These salts must be dried at 105° C. to a constant weight.

‡ Carnelley, Melting- and Boiling-point Tables.

§ Meyer, Riddle and Lamb, Ber. d. deut. Chem. Gesellsch., 27,

TABLE X.

GIVING THE NUMBER OF TIMES THE THEORETICAL QUANTITY OF AIR SUPPLIED, WITH VARIOUS GAS ANALYSES.\*

$\text{CO}_2 + \text{CO}$	N = 70. $\text{CO}_2 + \text{O} + \text{CO} = 21$	N = 80. $\text{CO}_2 + \text{O} + \text{CO} = 20$	N = 81. $\text{CO}_2 + \text{O} + \text{CO} = 19$	N = 82. $\text{CO}_2 + \text{O} + \text{CO} = 18$
21	1.00	....	....	....
20	1.05	1.00	....	....
19	1.10	1.05	1.00	....
18	1.17	1.10	1.05	1.00
17	1.23	1.16	1.10	1.05
16	1.31	1.23	1.16	1.10
15	1.40	1.31	1.23	1.16
14	1.50	1.39	1.30	1.22
13	1.61	1.49	1.39	1.30
12	1.75	1.60	1.48	1.38
11	1.91	1.73	1.59	1.47
10	2.10	1.89	1.72	1.58
9	2.33	2.07	1.87	1.70
8	2.62	2.29	2.04	1.85
7	3.00	2.57	2.26	2.02
6	3.50	2.92	2.52	2.23
5	4.20	3.39	2.86	2.48
4	5.25	4.05	3.30	2.79
3	7.00	5.00	3.89	3.20
2	10.50	6.53	4.76	3.76
1	21.00	9.43	6.10	4.54

\* Coxe, Proc. N. E. Cotton Manufacturers' Assoc., 1895.

TABLE XI.

COMPARISON OF METRIC AND ENGLISH SYSTEMS.

1 cubic inch = 16.39 c.c.  
 1 cubic foot = 28.315 liters.  
 1 Imperial gallon = 4.543 "

1 lb. avoirdupois = 453.593 grams.  
 1 calorie = 3.969 B.T.U. (Röntgen).

COAL AND FUEL OIL SPECIFICATIONS.

COAL SPECIFICATIONS.

The following, from Bulletin 339, U. S. Geological Survey (1908), by D. T. Randall, will give an idea of some coal specifications:

SPECIFICATIONS FOR THE U. S. GOVERNMENT FUEL SUPPLY AS APPROVED BY THE NATIONAL ADVISORY BOARD ON FUELS AND STRUCTURAL MATERIALS, MARCH, 1907.

SPECIFICATIONS AND PROPOSALS FOR SUPPLYING COAL.

United States.....  
....., 190.....

PROPOSAL.

Sealed proposals will be received at this office until 2 o'clock P.M.,..... 190.., for supplying coal to the United States.....building at..... as follows:

.....  
.....  
.....

The quantity of coal stated above is based upon the previous annual consumption, and proposals must be made upon the basis of a delivery of 10 per cent more or less than this amount, subject to the actual requirements of the service.

Proposals must be made on this form, and include all expenses incident to the delivery and stowage of the coal, which must be delivered in such quantities and at such times within the fiscal year ending June 30, 190.., as may be required.

Proposals must be accompanied by a deposit (certified check, when practicable, in favor of.....) amounting to 10 per cent of the aggregate amount of the bid submitted, as a guaranty that it is bona fide. Deposits will be returned to unsuccessful bidders immediately after award has been made, but the deposit of the successful bidder will be retained until after the coal shall have been delivered and final settlement made therefor, as security for the faithful performance of the terms of the contract, with the understanding that the whole or a part thereof may be used to liquidate the value of any

deficiencies in quality or delivery that may arise under the terms of the contract.

When the amount of the contract exceeds \$10,000, a bond may be executed in the sum of 25 per cent of the contract amount, and in this case the deposit or certified check submitted with the proposal will be returned after approval of the bond.

The bids will be opened in the presence of the bidders, their representatives, or such of them as may attend, at the time and place above specified.

In determining the award of the contract, consideration will be given to the quality of the coal offered by the bidder, as well as the price per ton, and should it appear to the best interests of the Government to award the contract for supplying coal at a price higher than that named in lower bid or bids received, the award will be so made.

The right to reject any or all bids and to waive defects is expressly reserved by the Government.

#### DESCRIPTION OF COAL DESIRED.\*

Bids are desired on coal described as follows:

.....  
 .....  
 .....

Coals containing more than the following percentages, based upon dry coal, will not be considered:

Ash.....	per cent
Volatile matter.....	per cent
Sulphur.....	per cent
Dust and fine coal as delivered at point of consumption †.....	per cent

#### DELIVERY

The coal shall be delivered in such quantities and at such times as the Government may direct.

In this connection it may be stated that all the available storage capacity of the coal bunkers will be placed at the disposal of the contractor to facilitate delivery of coal under favorable conditions.

---

\* This information will be given by the Government as may be determined by boiler and furnace equipment, operating conditions, and the local market.

† All coal which will pass through a  $\frac{1}{8}$ -inch round-hole screen.



After verbal or written notice has been given to deliver coal under this contract, a further notice may be served in writing upon the contractor to make delivery of the coal so ordered within twenty-four hours after receipt of said second notice.

Should the contractor, for any reason, fail to comply with the second request, the Government will be at liberty to buy coal in the open market, and to charge against the contractor any excess in price of coal so purchased over the contract price.

#### SAMPLING.

Samples of the coal delivered will be taken by a representative of the Government.

In all cases where it is practicable, the coal will be sampled at the time it is being delivered to the building. In case of small deliveries, it may be necessary to take these samples from the yards or bins. The sample taken will in no case be less than the total of 100 lbs., to be selected proportionally from the lumps and fine coal, in order that it will in every respect truly represent the quantity of coal under consideration.

In order to minimize the loss in the original moisture content the gross sample will be pulverized as rapidly as possible until none of the fragments exceed one-half inch in diameter. The fine coal will then be mixed thoroughly and divided into four equal parts. Opposite quarters will be thrown out, and the remaining portions thoroughly mixed and again quartered, throwing out opposite quarters as before. This process will be continued as rapidly as possible until the final sample is reduced to such amount that all of the final sample thus obtained will be contained in the shipping can or jar and sealed air-tight.

The sample will then be forwarded to.....

If desired by the coal contractor, permission will be given to him, or his representative, to be present and witness the quartering and preparation of the final sample to be forwarded to the Government laboratories.

Immediately on receipt of the sample, it will be analyzed and tested by the Government, following the method adopted by the American Chemical Society, and using a bomb calorimeter. A copy of the result will be mailed to the contractor upon the completion thereof.

## CAUSES FOR REJECTION.

A contract entered into under the terms of this specification shall not be binding if, as the result of a practical service test of reasonable duration, the coal fails to give satisfactory results owing to excessive clinkering or to a prohibitive amount of smoke.

It is understood that the coal delivered during the year will be of the same character as that specified by the contractor. It should, therefore, be supplied, as nearly as possible, from the same mine or group of mines.

Coal containing percentages of volatile matter, sulphur, and dust higher than the limits indicated on page 2 and coal containing a percentage of ash in excess of the maximum limits indicated in the following table will be subject to rejection.

In the case of coal which has been delivered and used for trial, or which has been consumed or remains on the premises at the time of the determination of its quality, payment will be made therefor at a reduced price, computed under the terms of this specification.

Occasional deliveries containing ash up to the percentage indicated in the column of "Maximum limits for ash," on page 4, may be accepted. Frequent or continued failure to maintain the standard established by the contractor, however, will be considered sufficient cause for cancellation of the contract.

## PRICE AND PAYMENT.\*

Payment will be made on the basis of the price named in the proposal for the coal specified therein, corrected for variations in heating value and ash, as shown by analysis, above and below the standard established by contractor in this proposal. For example, if the coal contains 2 per cent, more or less, British thermal units than the established standard, the price will be increased or decreased 2 per cent accordingly.

The price will also be further corrected for the percentages of ash. For all coal which by analysis contains less ash than that established

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\* The economic value of a fuel is affected by the actual amount of combustible matter it contains, as determined by its heating value shown in British thermal units per pound of fuel, and also by other factors, among which is its ash content. The ash content not only lowers the heating value and decreases the capacity of the furnace, but also materially increases the cost of handling the coal, the labor of firing, and the cost of removal of ashes, etc.

in this proposal a premium of 1 per cent per ton for each whole per cent less ash will be paid. An increase in the ash content of 2 per cent over the standard established by contractor will be tolerated without exacting a penalty for the excess of ash. When such excess exceeds 2 per cent above the standard established, deductions will be made from the price paid per ton in accordance with following table:

Ash as Established in Proposal (Per Cent).	No Deduction for Limits Below.	Cents per Ton to be Deducted.							Maximum Limits for Ash.
		2	4	7	12	18	25	35	
		Percentages of Ash in Dry Coal.							
5	7	7-8	8-9	9-10	10-11	11-12	12-13	13-14	12
6	8	8-9	9-10	10-11	11-12	12-13	13-14	14-15	13
7	9	9-10	10-11	11-12	12-13	13-14	14-15	15-16	14
8	10	10-11	11-12	12-13	13-14	14-15	15-16	16-17	14
9	11	11-12	12-13	13-14	14-15	15-16	16-17	17-18	15
10	12	12-13	13-14	14-15	15-16	16-17	17-18	.....	16
11	13	13-14	14-15	15-16	16-17	17-18	18-19	.....	16
12	14	14-15	15-16	16-17	17-18	18-19	19-20	.....	17
13	15	15-16	16-17	17-18	18-19	19-20	20-21	.....	18
14	16	16-17	17-18	18-19	19-20	20-21	21-22	.....	19
15	17	17-18	18-19	19-20	20-21	21-22	.....	.....	19
16	18	18-19	19-20	20-21	21-22	22-23	.....	.....	20
17	19	19-20	20-21	21-22	22-23	.....	.....	.....	21
18	20	20-21	21-22	22-23	.....	.....	.....	.....	22

Proposals to receive consideration must be submitted upon this form and contain all of the information requested.

.....  
 ....., 190..

The undersigned hereby agree to furnish to the U. S..... building at ....., the coal described, in tons of 2,240 lbs. each and in quantity 10 per cent more or less than that stated on page 1, as may be required during the fiscal year ending June 30, 190.., in strict accordance with this specification; the coal to be delivered in such quantities and at such times as the Government may direct.

	Item No...	Item No...	Item No...
<i>Description.</i>			
Commercial name.....			
Name of mine.....			
Location of mine.....			
Name of coal bed.....			
Size of coal (if coal is screened):			
Coal to pass through openings.....	.inches } round	.inches } round	.inches } round
Coal to pass over openings.....	.inches } square	.inches } square	.inches } square
	.inches } bar	.inches } bar	.inches } bar
<i>Data to establish a basis for payment.</i>			
Per cent of ash in dry coal (method of American Chemical Society).....			
British thermal units in coal as delivered.....			
Price per ton (2,240 lbs.) ..			

It is important that the above information does not establish a higher standard than can be actually maintained under the terms of the contract; and in this connection it should be noted that the small samples taken from the mine are invariably of higher quality than the coal actually delivered therefrom. It is evident, therefore, that it will be to the best interests of the contractor to furnish a correct description with average values of the coal offered, as a failure to maintain the standard established by contractor will result in deductions from the contract price, and may cause a cancellation of the contract, while deliveries of a coal of higher grade than quoted will be paid for at an increased price.

Signature.....

Address.....

Name of corporation.....

Name of president.....

Name of secretary.....

Under what law (State) corporation is organized.....

As will be seen from the foregoing specification, the bidder is not required to submit a sample of his coal, but is expected to name a standard of British thermal units in the coal as it is to be delivered. This value is made the basis for purchase, because a correction is thus made for the amount of moisture in the coal. It should be noted that this value will in all cases be lower than the British thermal units in the dry coal, which is usually given in connection with the coal analysis. The percentage of ash is also specified, as it is a factor in the successful burning of the coal on the grate and as it involves an expense for removal from the premises.



The following are the essential features of the contracts on which a Chicago company is said to purchase and inspect nearly 1,000,000 tons of coal for its clients in Chicago, Indianapolis, Minneapolis, St. Louis, and other cities:

I. The company agrees to furnish and deliver to the consumer . . . . .  
 . . . . . at such times and in such quantities as ordered by the consumer for consumption at said premises during the term hereof, at the consumer's option, either or all of the kinds of coal described below; said coals to average the following assays:

Kind of coal . . . . .	.....	.....	.....
Of size passing through screen having circular perforations in diameter . . . . .	..... inches	..... inches	..... inches
Of size passing over a screen having circular perforations in diameter . . . . .	..... inches	..... inches	..... inches
Per cent of moisture in coal as delivered . . . . .	.....	.....	.....
Per cent of ash in coal as delivered . . . . .	.....	.....	.....
British thermal units per pound of dry coal . . . . .	.....	.....	.....
From following county . . . . .	.....	.....	.....
From following State . . . . .	.....	.....	.....

Coal of the above respective descriptions and specified assays (not average assays) to be hereinafter known as the contract grade of the respective kinds.

II. The consumer agrees to purchase from the company all the coal required for consumption at said premises during the term of said contract, except as set forth in Paragraph III below, and to pay the company for each ton of 2000 lbs. avoirdupois of coal delivered and accepted in accordance with all the terms of this contract at the following contract rate per ton for coal of each respective contract grade, at which rates the company will deliver the following respective numbers of British thermal units for 1 cent, the contract guaranty:

Kind of Coal.	Contract Rate per Ton.	Contract Guaranty.
.....	\$ . . . . .	equal to . . . . . net B.T.U. for 1 cent
.....	\$ . . . . .	equal to . . . . . net B.T.U. for 1 cent
.....	\$ . . . . .	equal to . . . . . net B.T.U. for 1 cent

Said net British thermal units for 1 cent being in each case determined as follows: Multiply the number of British thermal units per pound of dry coal by the per cent of moisture (expressed in decimals),

subtract the product so found from the number of British thermal units per pound of dry coal, multiply the remainder by 2000, and divide this product by the contract rate per ton (expressed in cents) plus one-half of the ash percentage (expressed as cents).

III. It is provided that the consumer may purchase for consumption at said premises coal other than herein contracted for, for test purposes, it being understood that the total of such coal so purchased shall not exceed 5 per cent of the total consumption during the term of this contract.

IV. It is understood that the company may deliver coal hereunder containing as high as 3 per cent more ash and as high as 3 per cent more moisture and as low as 500 fewer British thermal units per pound dry than specified above for contract grades.

V. Should any coal delivered hereunder contain more than the per cent of ash or moisture or fewer than the number of British thermal units per pound dry allowed under Paragraph IV hereof, the consumer may, at its option, either accept or reject same.

VI. All coal accepted hereunder shall be paid for monthly at a price per ton determined by taking the average of the delivered values obtained from the analyses of all the samples taken during that month, said delivered value in each case being obtained as follows: Multiply the number of British thermal units delivered per pound of dry coal by the per cent of moisture delivered (expressed in decimals), subtract the product so found from the number of British thermal units delivered per pound of dry coal, multiply the remainder by 2000, divide this product by the contract guaranty, and from this quotient (expressed as dollars and cents) subtract one-half of the ash percentage delivered (expressed as cents).

---

The following are the essential features of the specifications used by the Interborough Rapid Transit Company of New York in purchasing about 30,000 tons of coal each month for use in its plants, which are among the largest in the United States:

PRELIMINARY SPECIFICATIONS FOR BITUMINOUS COAL FOR THE INTERBOROUGH RAPID TRANSIT COMPANY.

Coal must be a good steam, caking, run-of-mine, bituminous coal free from all dirt and excessive dust, a dry sample of which will approximate the company's standard in heat value and analysis as follows: Carbon, 71; volatile matter, 20; ash, 9; B.T.U., 14,100; sulphur, 1.50.

A small quantity of coal will be taken from each weighing hopper just before the hopper is dumped while the lighter is being unloaded. These quantities will be thrown into a receptacle provided for the purpose, and when the lighter is empty the contents of the receptacle will be thoroughly mixed, and a sample of this mixture will be taken for chemical analysis. This average sample of coal will be labeled and held for one week after the unloading of the lighter. The sample taken from the mixture for test will be analyzed as soon as possible after being taken. No other sample will be recognized.

Tests of sample taken from average sample will be made by the company's chemist under the supervision of the superintendent. Should the contractor question the results of the company's test (a copy of which will be mailed to him), the company will, if requested by the contractor within three days after copy of test has been mailed to him, forward sufficient quantity of the average sample taken from each weighing hopper to any laboratory in the city of New York which may be agreed upon by the superintendent and the contractor, and have said sample analyzed by it, and the results obtained from this second test will be considered as final and conclusive. In case the disputed values, as obtained in the company's test, shall be found by the second test to be 2 per cent or less in error, then the cost of said second test shall be borne by the contractor; but if the disputed values shall be found to be more than 2 per cent in error, then the cost of said second test shall be borne by the company.

Should there be no question raised by the contractor within the three days specified, as to the values of the first analysis, the average sample of coal will be destroyed at the end of seven days from date of discharge of coal from lighter. Should a second test be made of coal taken from any lighter as herein provided, then any penalties to be made as set forth in paragraph under "Penalties" will be based on the results as obtained from the second test.

The price to be paid by the company per ton per lighter of coal will be based on a table of heat values for excess or deficiency of its standard, but subject to deductions as given in the section under "Penalized coal," including excess of ash, volatile matter, sulphur, or dust, or less than the minimum amount required to be contained in any lighter, for coal which shows results less than the company's standard.

Premiums or deductions are based on a rate of 1 cent per ton for a variation of 50 B.T.U. per pound of coal, as indicated in a table a few items of which are given below:

*Table for B.T.U. Values.*

For coal in any lighter which is found by test to contain, per pound of dry coal, from

15,501 and above . . . . .	28 cents per ton above standard
15,101 to 15,150, both inclusive . . . . .	20 cents per ton above standard
14,601 to 14,650, both inclusive . . . . .	10 cents per ton above standard
14,101 to 14,150, both inclusive . . . . .	Standard
13,601 to 13,650, both inclusive . . . . .	10 cents per ton below standard
13,101 to 13,150, both inclusive . . . . .	20 cents per ton below standard
12,101 to 12,150, both inclusive . . . . .	40 cents per ton below standard

No lighter of coal will be accepted which, by trial, in the judgment of the superintendent, contains an excessive amount of dry coal dust. The decision of the superintendent will be final in this respect. Coal taken from such lighter for trial will be subject to the special deduction set forth under "Penalized coal," but paid for in all other respects as herein provided.

Coal which is shown by analysis to contain less than 20 per cent of volatile matter, 9 per cent of ash, or 1.50 per cent of sulphur, will be accepted, without a deduction from the bidder's price, plus or minus an amount for excess or deficiency of British thermal unit value, as herein provided. Where the analysis gives amounts for any or all elements in excess of these quantities, deductions will be made from the bidder's price in accordance with the tables of values of volatile matter, ash, and sulphur below given, plus or minus the amount for excess or deficiency of the standard British thermal unit value, in addition to any other deductions which may be made as herein provided.

*Table of Deductions for Volatile Matter.*

For coal in any lighter which is found by test to contain, per pound of dry coal

Over 20 per cent and less than 21 per cent . . . . .	2 cents per ton
* * * * *	* * * * *
Over 22.5 per cent and less than 23 per cent . . . . .	12 cents per ton
* * * * *	* * * * *
24 per cent and over . . . . .	18 cents per ton

This table is made for the difference of each one-half of 1 per cent and the deductions are at the rate of 4 cents for each 1 per cent of volatile matter.



*Table of Deductions for Ash.*

For coal in any lighter which is found by test to contain, per pound of dry coal

Over 9 per cent and less than 9.5 per cent . . . . .	2 cents per ton
* * * * *	* * * * *
Over 11.5 per cent and less than 12 . . . . .	12 cents per ton
* * * * *	* * * * *
13.5 per cent and over . . . . .	23 cents per ton

This table is made for each difference of one-half of 1 per cent and at the rate of 4 cents for each 1 per cent increase in the ash.

*Table of Deductions for Sulphur.*

For coal in any lighter which is found by test to contain, per pound of dry coal

Over 1.50 per cent and less than 1.75 per cent . . . . .	6 cents per ton
* * * * *	* * * * *
Over 2 per cent and less than 2.25 per cent . . . . .	10 cents per ton
* * * * *	* * * * *
2.50 and over . . . . .	20 cents per ton

This table is made out for each difference of one-fourth of 1 per cent and at a diminishing rate.

Should any lighter of coal delivered at the company's docks contain less than 700 tons, a deduction of 7 cents per ton will be made from the price as determined by the British thermal unit value and analysis, in addition to any other penalty provided for herein. Should any lighter of coal delivered at the company's docks be rejected by the superintendent on account of excessive amount of coal dust, then a reduction of 25 cents per ton will be made from the price as determined by the British thermal unit value and analysis, for the coal taken from said lighter, in addition to any other penalty which may be made as herein provided. Should any lighter of coal be delivered in other than self-trimming lighters as herein provided, a deduction of 7 cents per ton will be made from the price as determined by the British thermal unit value and analysis, exclusive of any other penalty which may be made as herein provided.

The contractor's bill of lading will be checked by the company's scales. Should there be a deficiency of 1 per cent or more between

the bill of lading and the company's weights, then the company's weights will be taken as correct.

When the contractor has been notified by the company to deliver coal under this contract, a further notice may be given requiring the contractor to make delivery of the coal so ordered within twelve hours after the receipt of said second notice. Should the contractor, for any reason, fail to deliver the coal so ordered within twelve hours after the receipt of said second notice and in accordance with the requirements therein as to place of delivery, the company shall be at liberty to buy coal in the open market, and the contractor will make good to the company any difference there may be between the price paid by the company for said coal in open market and the price the company would have paid to the contractor had the coal been delivered by it in accordance with the requirements of said notices from the company, or said difference may be deducted from any money then due or thereafter to become due to the contractor under the contract to be entered into.

#### FUEL OIL SPECIFICATIONS.

The Specifications of the U. S. Government are as follows\*:

##### GENERAL SPECIFICATIONS.

(1) In determining the award of a contract, consideration will be given to the quality of the fuel offered by the bidders, as well as the price, and should it appear to be to the best interest of the Government to award a contract at a higher price than that named in the lowest bid or bids received, the contract will be so awarded.

(2) Fuel oil should be either a natural homogeneous oil or a homogeneous residue from a natural oil; if the latter, all constituents having a low flash-point should have been removed by distillation; it should not be composed of a light oil and a heavy residue mixed in such proportions as to give the density desired.

(3) It should not have been distilled at a temperature high enough to burn it, nor at a temperature so high that flecks of carbonaceous matter began to separate.

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\* J. C. Allen, *J. Ind. and Eng. Chem.*, 3, 730 (1911).

(4) It should not flash below 60° C. (140° F.) in a closed Abel-Pensky or Pensky-Martens tester.

(5) Its specific gravity should range from 0.85 to 0.96 at 15° C. (59° F.); the oil should be rejected if its specific gravity is above 0.97 at that temperature.

(6) It should be mobile, free from solid or semi-solid bodies, and should flow readily, at ordinary atmospheric temperatures and under a head of 1 foot of oil, through a 4-inch pipe 10 feet in length.

(7) It should not congeal nor become too sluggish to flow at 0° C. (32° F.).

(8) It should have a calorific value of not less than 10,000 calories per gram \* (18,000 B.T.U. per pound), 10,250 calories to be the standard. A bonus is to be paid or a penalty deducted according to the method stated under Section 21, as the fuel oil delivered is above or below this standard.†

(9) It should be rejected if it contains more than 2 per cent water.

(10) It should be rejected if it contains more than 1 per cent sulphur.

(11) It should not contain more than a trace of sand, clay, or dirt.

(12) Each bidder must submit an accurate statement regarding the fuel oil he proposes to furnish. This statement should show:

(a) The commercial name of the oil.

(b) The name or designation of the field from which the oil is obtained.

(c) Whether the oil is a crude oil, a refinery residue, or a distillate.

(d) The name and location of the refinery, if the oil has been refined at all.

(13) The fuel oil is to be delivered f.o.b. cars or vessel, according to the manner of shipment, at such places, at such times, and in such quantities as may be required, during the fiscal year ending. . . . .

(14) Should the contractor, for any reason, fail to comply with a written order to make delivery, the Government is to be at liberty to buy oil in the open market, and charge against the contractor any excess of price, above the contract price, of the fuel oil so purchased.

---

\* Calories  $\times 1.8 =$  B.T.U. per pound.

† It is important that the standard fixed should not be higher than can be maintained under the terms of the contract. In the absence of information as to the heating value of the oil, the Bureau of Mines will analyze samples taken from the deliveries to establish the standard heating value, expressed in calories or B.T.U. It will be to the best interests of the contractor to specify a fair standard for the fuel oil he offers, since failure to maintain that standard will cause deduction from the contract price and possibly the cancellation of the contract, while deliveries of higher quality than the standard will result in the contractor receiving premiums.

## SAMPLING.

(15) Deliveries of fuel oil will be sampled by a representative of the Government. Whenever such action is practicable, the oil will be sampled as it is being delivered. The final sample will be made from samples taken from as large a proportion of the delivery as practicable, in order that the final sample may truly represent the delivery.

(16) The final sample will be sealed and forwarded to the Federal Bureau of Mines, Pittsburgh, Pa., for analysis:

(17) If the contractor so desires, permission will be given him, or his representative, to witness the sampling of the delivery and the preparation of the final sample.

(18) The final sample will be analyzed and tested immediately after its receipt in Pittsburgh.

## CAUSES FOR REJECTION.

(19) A contract entered into under the terms of these specifications shall not be binding if, as the result of a practical service test of reasonable duration, the fuel oil fails to give satisfactory results.

(20) It is understood that the fuel oil delivered during the terms of the contract shall be of the quality specified. The frequent or continued failure of the contractor to deliver oil of the specified quality will be considered sufficient cause for the cancellation of the contract.

## PRICE AND PAYMENT.

(21) Payment for deliveries will be made on the basis of the price named in the proposal for the fuel oil corrected for variations in heating value,\* as shown by analysis, above or below the standard fixed by the contractor. This correction is a *pro rata* one and the price is to be determined by the following formulæ:

$$\frac{\text{Delivered calories per gram (or B.T.U. per lb.)} \times \text{contract price}}{\text{Standard calories per gram (or B.T.U. per lb.)}} = \text{price to be paid.}$$

---

\* The value of an oil as fuel is in proportion to the total combustible matter it contains as shown by its heating value. This value may be expressed in small calories per gram of B.T.U. per pound. Sulphur, moisture, and earthy matter lower the heating value of an oil and decrease the furnace capacity; they also may have a deleterious effect on boiler and furnace, and may impair the operation of burners.



Water that accumulates in the receiving tank will be drawn off and measured periodically. Proper deduction will be made by subtracting the weight of the water from the weight of the oil deliveries.

DETERMINATION OF WEIGHT FROM VOLUME.

The specifications given on the preceding pages provide for the purchase of fuel oil by weight. As such oil is frequently delivered by volume, it is important to note the temperature of a delivery and to allow for the expansion due to this temperature when computing the weight of the delivery from the volume. From the volume of the oil at the temperature of delivery, the volume at standard temperature (15° C.) should be computed in the manner given below.

The coefficient of expansion of ordinary fuel oil residues of asphaltic base is approximately 0.0006 per 1° C.

Hence if the temperature ( $N^{\circ}$  C.) of the delivery is above 15° C., then  $(N^{\circ}$  C. - 15° C.)  $\times$  0.0006 = correction.

This correction is to be added to the specific gravity at  $N^{\circ}$  C. to give the standard specific gravity, that at 15° C.

If the temperature ( $N^{\circ}$  C.) of the oil delivered is below 15° C., the correction  $((15^{\circ}$  C. -  $N^{\circ}$  C.)  $\times$  0.0006) is to be subtracted from the specific gravity at 15° C.

Since a gallon of water at a temperature of 15° C. weighs 8.3316 lbs., the weight in pounds of a gallon of oil at 15° C. is 8.3316 times the specific gravity of the oil at that temperature.

Similarly, since a cubic foot of water at 15° C. weighs 62.3425 lbs., the weight in pounds of a cubic foot of oil at 15° C. is 62.3425 times its specific gravity at that temperature.

REPORTING ANALYSES OF FUEL OIL.

The following form is used by the Bureau of Mines in reporting the results of an analysis of a sample of fuel oil:

DEPARTMENT OF THE INTERIOR.

BUREAU OF MINES.

WASHINGTON, D. C., ..... 191—.

SIR:

In reference to the ..... sample of fuel petroleum represent-  
 (Quantity.)  
 ing ..... of petroleum delivered at a temperature of ...° C. by  
 (Quantity.)  
 the ..... as a ..... product, from the .....  
 (Company delivering.) (Crude, residue, or distillate.) (Lease.)

....., to the .....  
 (Field or district.) (County.) (State.) (Department receiving.)  
 at ..... on ....., I have the honor to report as  
 (City.) (Date of delivery.)  
 follows:

- Specific gravity at 15° C. ....
- (Baumé at 59° F.).....
- Calorie per gram.....
- (B.T.U. per pound).....
- Water, per cent. ....
- Sulphur, per cent.....
- Earthy matter, sand, etc., per cent.....
- Flash-point, ° C. (Abel-Pensky, or Pensky-Mar-  
 tens, closed tester).....
- Burning point, ° C. (same tester, opened).....
- Remarks:

.....  
 .....

---

The above information is for the use of the Government and the dealer or operator furnishing the oil. It is to be considered confidential until it is published by the United States Government.

---

Respectfully,

.....

*Chief Clerk.*

Certified:

.....

*Petroleum Chemist.*

### SAMPLING PETROLEUM OR FUEL OIL.

#### GENERAL STATEMENT.

The accuracy of the sampling and, in turn, the value of the analysis must necessarily depend on the integrity, alertness, and ability of the person who does the sampling. No matter how honest the sampler may be, if he lacks alertness and sampling ability, he may easily make errors that will vitiate all subsequent work and render the results of tests and analyses utterly misleading. A sampler must be always on the alert for sand, water, and foreign matter. He should note any circumstances that appear suspicious, and should submit a critical report on them, together with samples of the questioned oil.

## SAMPLING WAGON DELIVERIES.

## SAMPLING WITH A DIPPER.

Immediately after the oil begins to flow from the wagon to the receiving tank, a small dipper holding any definite quantity, say, 0.5 liter (about 1 pint), is filled from the stream of oil. Similar samples are taken at equal intervals of time from the beginning to the end of the flow—a dozen or more dipperfuls in all. These samples are poured into a clean drum and well shaken. If the oil is heavy, the dipperfuls of oil may be poured into a clean pail and thoroughly stirred. For a complete analysis the final sample should contain at least 4 liters (about 1 gallon). This sample should be poured into a clean can, soldered tight and forwarded to the laboratory.

It is important that the dipper be filled with oil at uniform intervals of time and that the dipper be always filled to the same level. The total quantity of oil taken should represent a definite quantity of oil delivered and the relation of the sample to the delivery should be always be stated, for instance: "1 gallon sample representing 1 wagon-load of 20 barrels."

## CONTINUOUS SAMPLING.

Instead of taking samples with a dipper, it may be more convenient to take a continuous sample. This may be taken by allowing the oil to flow at a constant and uninterrupted rate from a  $\frac{1}{2}$ -inch cock on the under side of the delivery pipe during the entire time of discharge. The continuous sample should be thoroughly mixed in a clean drum or pail, and at least 4 liters (about 1 gallon) of it forwarded for analysis. A careful examination should be made for water, and if the first dipperful shows water this dipperful should be thrown into the receiving tank and not mixed with the sample for analysis.

## MIXED SAMPLES.

If the oil delivered during any definite period of time, say one month, be from the same source and of uniform quality (but only in case it is of uniform quality), it may suffice to pour definite proportional quantities of the dipper and the continuous samples taken during this period into a tinned can or drum having a tight screw cap or bung. An iron drum should not be used, since even a clean iron surface will absorb sulphur by long contact with a sulphur-containing oil, and this sulphur will be lost to the analyst. At the end of the month a number of round,

clean stones should be put into the drum and the drum should be rolled to insure intimate mixing. Then 4 liters (about 1 gallon) of the gross sample should be taken for analysis. The drum should be drained, rinsed clean with gasoline, dried, and made ready for a second sampling.

The all-important point is that the gross sample, whatever the manner of sampling, shall be made up of equivalent portions of oil taken at regular intervals of time, so that the sample finally forwarded for analysis will truly represent the entire shipment.

#### SAMPLING A LARGE TANK OR RESERVOIR.

Water or earthy matter settles on standing. Hence, before a large stationary tank or reservoir is sampled the character of the contents at the bottom should be ascertained by dredging with a long-handled dipper, and the content of the dipper should be examined critically. If a considerable quantity of sediment is brought up, it should be cause for rejecting the oil.

The sampling of a large stationary tank or reservoir of oil, particularly if the oil has stood so long that it has begun to stratify, or form layers of different density, may be done as follows:

The sampler should procure an ordinary iron pipe, or preferably a tinned tube, 1 inch in diameter and long enough to reach from above the manhole, where he can grasp it, to the bottom of the tank. The lower end of the pipe should be reamed out with a round file. A conical plug of cork, wood, or other suitable material should be fitted to this end, and a strong, stiff wire, such as the ordinary telegraph wire, run through this plug and up through the pipe to a point where it can be grasped firmly by the sampler. A pull on the wire will close the bottom of the pipe, and a rap against the bottom of the tank will drive the plug home and make an oil-tight seal or valve.

To operate this sampling device, the sampler should remove the plug, allow it to drop some three inches below the bottom of the pipe, and let it hang there by the wire extending above the pipe. Then holding the pipe, open at top and bottom, in a vertical position, the sampler should allow it to sink slowly through the oil to the bottom of the tank. He should do this slowly and with care, so that the pipe will penetrate the oil without agitating it and will thus cut a representative core of oil from the surface to the bottom. When the pipe touches the bottom, the sampler should draw up the slack of the wire and pull the plug into place; then he should strike the plug smartly against the bottom of the tank, thereby driving it home and sealing



the pipe. He can then withdraw the pipe and pour the oil into the sampling can. If it seems desirable, he should "core" or "sample" a reservoir at regularly spaced points, unite these samples, mix them thoroughly, and take 4 liters (about 1 gallon) of the gross sample for analysis.

Instead of a pipe sampler, a bottle holding half a liter (about 1 pint) may be used. It should be securely fastened to a long pole and have a loosely-fitted stopper tied to a strong cord. The bottle, corked and empty, is immersed to any desired point within the mass of oil, and the stopper is pulled out. The bottleful of oil is poured into a suitable receiving vessel, and the bottle thoroughly drained is made ready for a second filling. Bottlefuls of oil taken in this way from points symmetrically placed throughout the mass of oil, will, if properly mixed, provide an excellent gross sample from which to take the 4-liter (1 gallon) sample for analysis.

#### SAMPLING A SINGLE DRUM.

A single drum may be sampled with a glass tube. This tube, open at both ends, should be grasped at the top, held vertically, inserted in the drum without agitating the oil, and allowed to cut its way slowly to the bottom of the drum. The upper end should then be closed with the thumb or forefinger of the hand holding it, the tube withdrawn, and the oil on the outside wiped off with the fingers of the other hand. The sample in the tube can then be transferred to a small can, and forwarded for analysis.

#### FORWARDING SAMPLES.

The sample should be forwarded in a glass bottle or carboy or in a tin can, preferably in the latter, because less liable to breakage. If a tin can is used the cap should be soldered tight. The can should not be filled completely; about an eighth of an inch of space should be left to allow for possible expansion of the oil.

The can should be sealed as soon as it is filled to avoid loss by volatilization of the lighter constituents of the sample. After the can has been filled and tightly soldered, it should be wiped clean and carefully examined for pinholes or small leaks. All leaks should be soldered before the can is packed for shipment.

The bottle or can should be carefully labeled. The following form of label,\* used by the Bureau of Mines, should be placed on samples shipped to the bureau:

---

\* These labels will be furnished on request.



## SAMPLING GAS FROM A WELL.

Since the gas associated with oil is an ideal fuel and illuminant, and the literature dealing with the composition of natural gas is scanty, a description of the method of sampling such gas for analysis is here given.

For taking a sample of gas under pressure from an oil well a cloth funnel should be made by folding and sewing any strong, closely-woven cloth into the form of a cornucopia. The larger end of this funnel should be large enough to encompass the gas pipe from which the sample is to be taken. The smaller end, or apex, of the funnel should be securely tied about one end of a flexible rubber tube 1 or 2 feet long and one-fourth to one-half inch in diameter. If there is a gas jet at the well, one end of the rubber tube may be attached directly to the jet.

A gas-sampling bottle should be procured, if practicable, from the Bureau of Mines, Pittsburgh, Pa. If such a bottle is not at hand, a 1- or 2-liter (1- or 2-quart) bottle with a well-ground, tight-fitting glass stopper may be used. The bottle should be thoroughly cleansed and dried. A large perfume bottle or an acid bottle, such as may be obtained from a drug store, will usually answer. A glass stopper is essential, for a cork or rubber stopper may leak even though it appears to be hermetically sealed with wax; moreover, a cork or rubber stopper may contaminate the gas.

To collect a sample, the funnel should be tied firmly about the end of the gas pipe. The funnel and the rubber tube should then be thoroughly flushed with the gas to rid them of air. The free end of the tube should go to the bottom of the sample bottle. The bottle should be fastened bottom up and the gas allowed to blow strongly into it for at least a quarter of an hour to insure complete expulsion of air. If the gas pressure is low, the gas should be allowed to blow longer, or until it is certain that all air has been expelled from the bottle. Meanwhile the stopper of the bottle should have been well greased with vaseline.

While the gas is still blowing through the tube the tube should be slowly withdrawn. The stopper should be put in just as soon as the tube is withdrawn and should be turned firmly into place. Then the bottle should be turned up and a spoonful of melted paraffin poured over the stopper. The stopper should be secured with elastic band.

A strong tag should be tied to the bottle by a stout cord. This tag should be labeled as follows:

*Gas Sample.*

Sampled by.....

Date.....

Well..... Lease.....

Number.

Section.....Township.....Range.....

District.....County.....State.....

Remarks.....

.....

.....

The bottle should be packed securely in a box and forwarded to the Bureau of Mines, Pittsburgh, Pa. A duplicate copy of the label should be sent to the same address.



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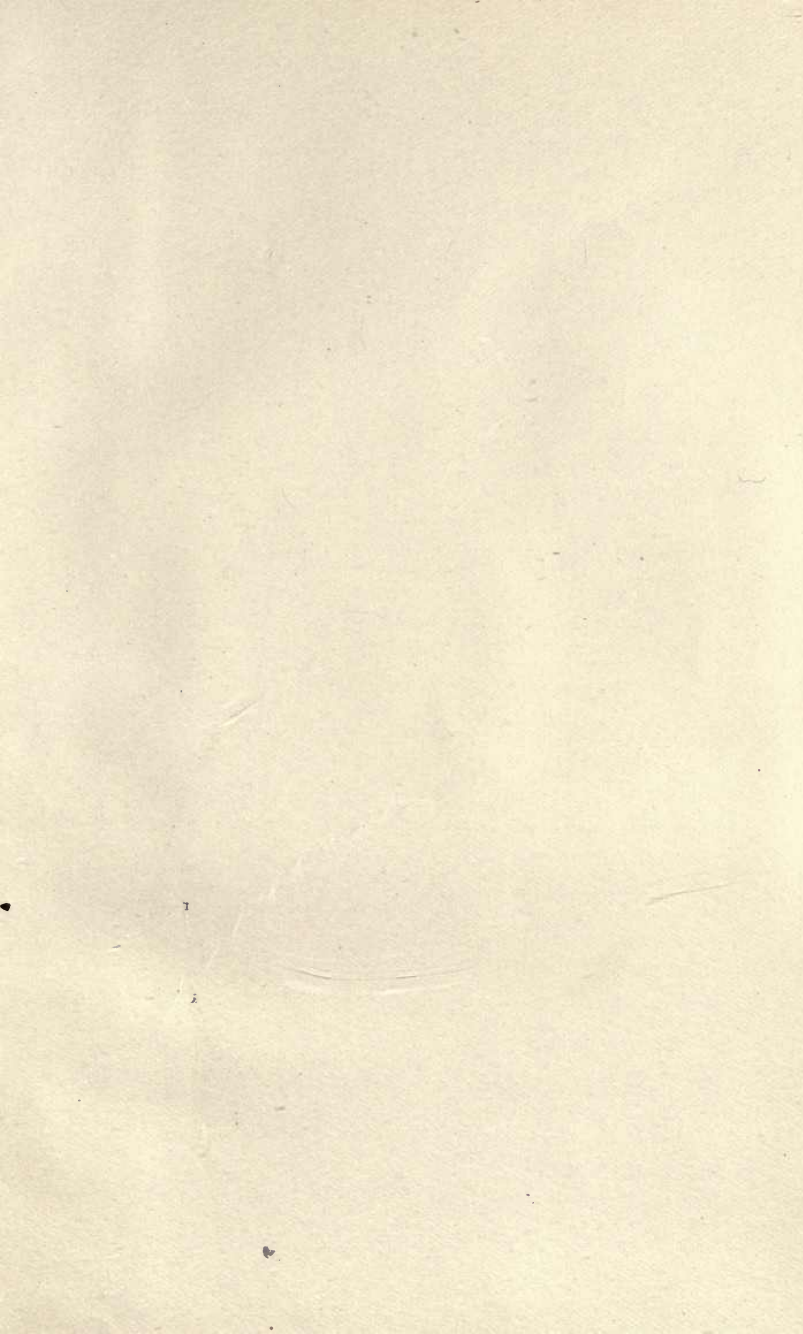


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