

# ELEMENTARY APPLIED CHEMISTRY

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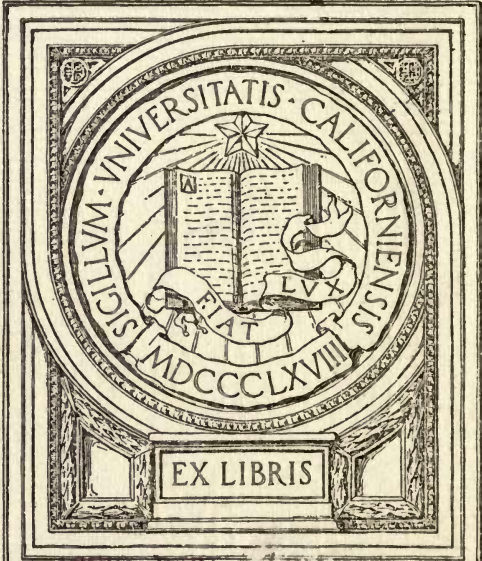
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LEWIS B. ALLYN

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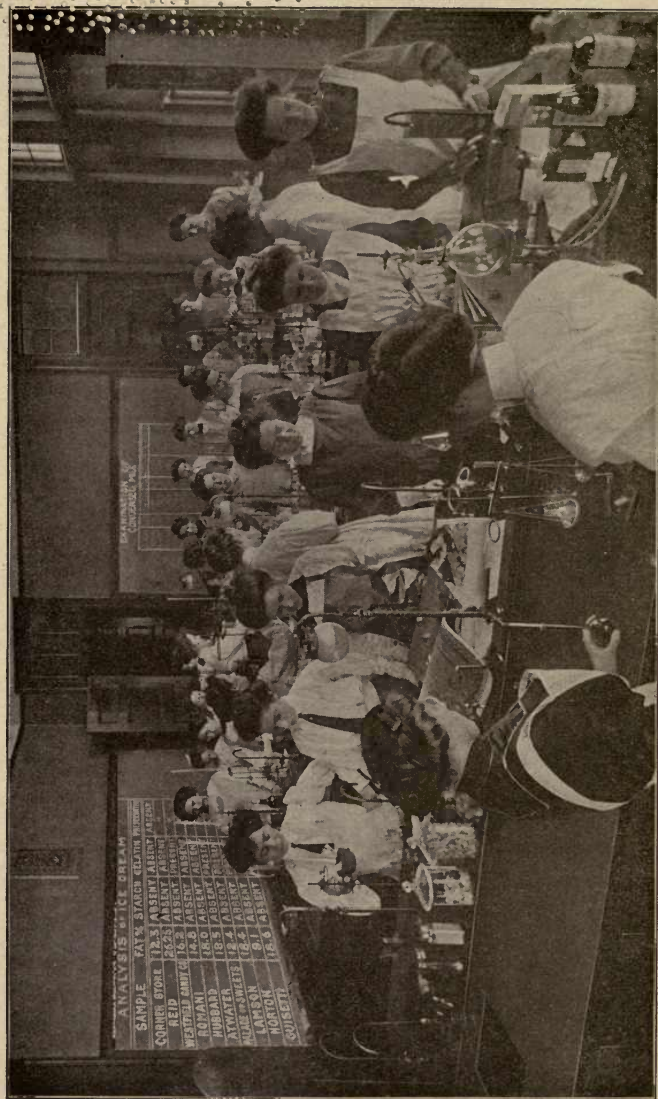


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The Three I's of Chemistry — Interest, Industry, Individuality

# ELEMENTARY APPLIED CHEMISTRY

BY

LEWIS B. ALLYN

DEPARTMENT OF CHEMISTRY, STATE NORMAL SCHOOL  
WESTFIELD, MASSACHUSETTS

"For the world was built in order  
And the atoms march in tune."

—EMERSON.



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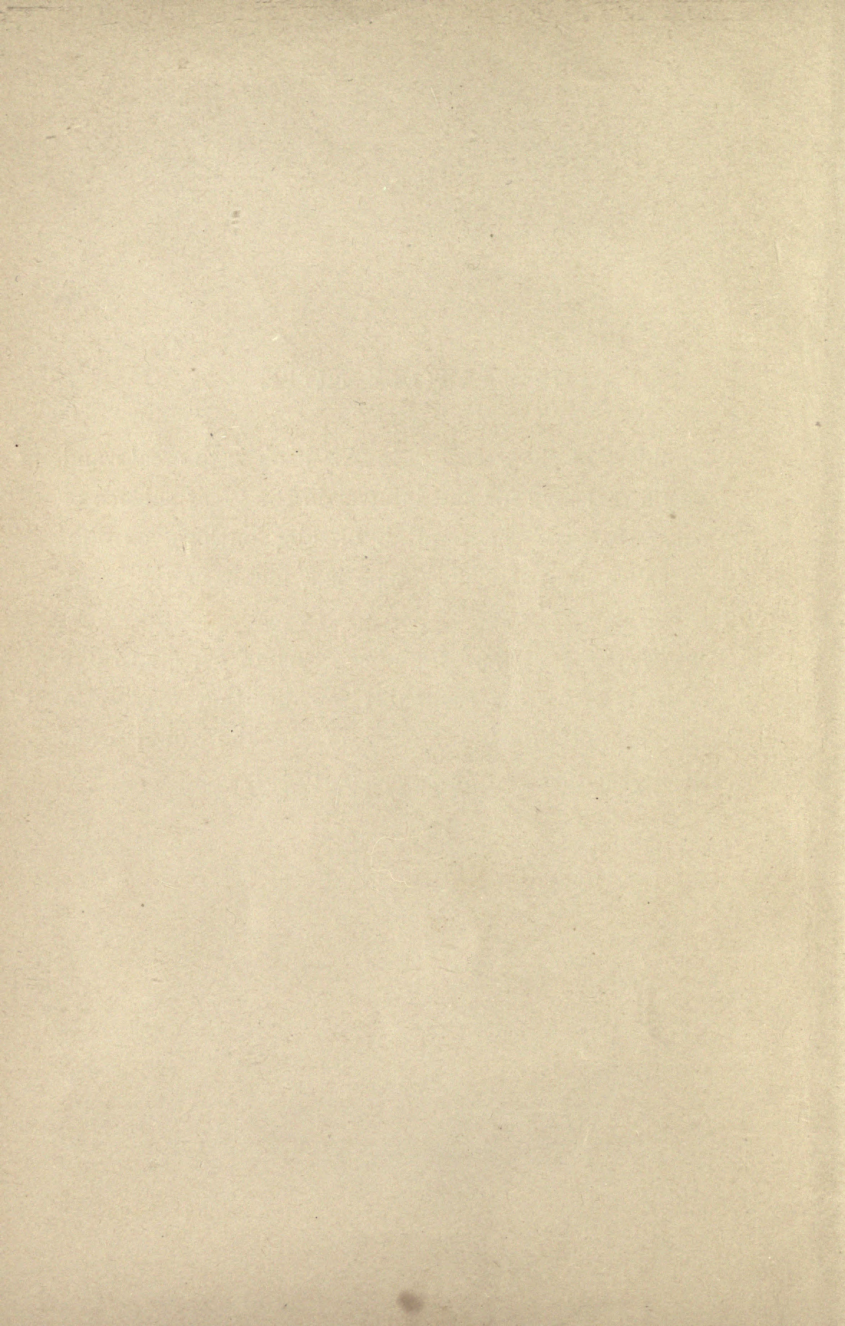
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## PREFATORY NOTE

The object of the exercises in this book is to create and to foster a real love for and interest in the great science of chemistry, to give the pupil a broader outlook on life, and to cause him to feel that he is a factor in the busy, living world.

These experiments and tests have been of personal value to hundreds of earnest students; possibly they may be of value to you. If you know of some one who would profit by the information you may receive, pass it on.

445101





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## SUGGESTIONS TO TEACHER AND PUPIL

Be sure that the work done is correct, and then certify it.

The use of blanks similar to the one on page x gives a suggestion of importance to the task performed and is conducive to honest, accurate work, and, best of all, to a regard for the truth.

Much time will be saved by making counterpoises with stoppered vials and fine shot for beakers, crucibles, evaporating dishes and specific-gravity flasks. Test the accuracy of these tares occasionally.

If you are not sure of a reaction, work with a sample which is known to contain the substance in question. For example, if the odor of phenylcarbamine is unknown, heat a few drops of commercial anilin with 5 cc. of stock solution of KOH. Add a cubic centimeter of chloroform, agitate gently, cool, and note the odor.

Form the habit of using a pipette instead of a graduate whenever special accuracy is desired.

Distilled water should be used for dilutions and solutions, especially for solutions of precision.

Many semifluid substances may be ashed by allowing the flame to strike the surface of the material, meanwhile applying a gentle heat to the bottom of the crucible.

Save products and samples. Bottle and label them. A collection is an inspiration and an incentive.

Notebooks should be illustrated by mounted samples, cuts, and clippings from newspapers and magazines.

## REPORT OF CHEMICAL ANALYSIS

---



---

**Sample** \_\_\_\_\_

**Obtained from** \_\_\_\_\_

**Manufactured by** \_\_\_\_\_

|                                     |       |
|-------------------------------------|-------|
| Reaction . . . . .                  | _____ |
| Test . . . . .                      | _____ |
| Coal-tar dye . . . . .              | _____ |
| Vegetable colors . . . . .          | _____ |
| Alcohol { ethyl . . . . .           | _____ |
| { methyl . . . . .                  | _____ |
| Mineral matter or metals . . . . .  | _____ |
| Organic compounds (miscellaneous) . | _____ |
| Bases . . . . .                     | _____ |
| Radicals . . . . .                  | _____ |
| Essential oils . . . . .            | _____ |
| Preservatives . . . . .             | _____ |
| Glucose . . . . .                   | _____ |
| Acetanilid or phenacetin . . . . .  | _____ |
| Equation . . . . .                  | _____ |
| Adulteration { fraudulent . . . . . | _____ |
| { injurious . . . . .               | _____ |
| Quality . . . . .                   | _____ |

## REMARKS

---



---

 I hereby certify that the above is correct to the best of my knowledge.

Name \_\_\_\_\_

Date \_\_\_\_\_ 19\_\_\_\_

Much collateral reading should be encouraged. The following books and pamphlets are valuable:

- LEACH, A. E. Food Inspection and Analysis. John Wiley & Sons.  
WILEY, H. W. Foods and their Adulteration. Blakiston.  
BLYTH, A. W. Foods, their Composition and Analysis. D. Van  
Nostrand Company.  
PEARSON. Jensen's Milk Hygiene. J. B. Lippincott.  
OLSEN, J. C. Pure Foods. Ginn and Company.  
OLSEN, J. C. Quantitative Chemical Analysis. D. Van Nostrand  
Company.  
SNYDER. Human Foods. The Macmillan Company.  
ALLEN, A. H. Commercial Organic Analysis. D. Van Nostrand  
Company.  
COHN. Tests and Reagents. John Wiley & Sons.  
*Bulletin No. 107*, Bureau of Chemistry, United States Department  
of Agriculture.



# ELEMENTARY APPLIED CHEMISTRY

## SECTION I

### FILTRATION

**Application to Qualitative and Quantitative Analysis (2-Part Compound).** A very satisfactory remedy for tonsillitis is a mixture consisting of equal parts of sulfur and powdered sugar. This is a recognized specific, and at one time was sold as a patent medicine at fifty cents per ounce. The instructor should prepare a quantity of the specific, varying the proportions slightly. Pass the mixture several times through a fine sieve.

(a) *Qualitative Work.* Place a spoonful of the powder in a beaker. Add 50 cc. of water, boil, and filter. Test both filtrate and residue in any way you choose. Of what is the powder composed? How do you know?

Copy and sign the following statement:

I hereby certify that a mixture called Tonsillitis Specific and examined by me contains \_\_\_\_\_.

Name \_\_\_\_\_

Date \_\_\_\_\_

(b) *Quantitative Work.* Weigh as exactly as possible 3 to 5 g. of the specific upon a carefully balanced filter paper. Adjust to a funnel and wash with repeated portions of hot water until the filtrate ceases to darken when a few

## 2 ELEMENTARY APPLIED CHEMISTRY

drops are heated with strong  $H_2SO_4$ ; or until a drop leaves no dark-colored residue when evaporated upon a piece of platinum foil.

Dry the residue over a water bath; remove and cool.

Place the duplicate filter paper in the opposite scale pan and weigh the sulfur directly. Determine the weight of the sugar by difference.

Copy and sign the following, or use a printed blank:

The sample of Tonsillitis Specific as analyzed by me contains \_\_\_\_\_ per cent sulfur, \_\_\_\_\_ per cent sugar.

I hereby certify that the above is correct to the best of my knowledge.

Name \_\_\_\_\_

**Application to the Analysis of a 3-Part Compound.** (a) Introduce a definite amount of gunpowder (2 to 3 g.) into a balanced filter paper. Wash with repeated portions of hot water. Test the filtrate for residue with the platinum foil as before, using only a drop or so for the test. Collect the filtrate and evaporate it to dryness in a tared evaporating dish.

(b) Dry the residue remaining in the filter paper. *Extinguish all flame* in the vicinity, and wash the residue with three or four 10-cc. portions of  $CS_2$ . Collect filtrate No. 2 in a small beaker. Blow on it gently through a pointed tube until evaporation is complete, or allow it to evaporate spontaneously. What remains?

Dry the black residue over a water bath and weigh with the counterpoised filter.

Record and certify your results.

Character and per cent of the white salt . . . \_\_\_\_

Character and per cent of the black residue . . . \_\_\_\_

Character and per cent of the yellow substance . \_\_\_\_

Read on the history and composition of gunpowder.



**Application to Quantitative Analysis of Soil.** Thoroughly dry about 5 g. of soil procured by the pupil. Weigh exactly upon a balanced and folded filter paper. Place in a funnel and wash with successive portions of hot water until the filtrate does not darken when heated with an equal volume of concentrated  $H_2SO_4$ . Dry the filter paper and contents at  $100^\circ C$ . Reweigh and calculate the loss of weight as soluble matter. Transfer the dried residue to a counterpoised porcelain or quartz crucible and determine its exact weight. Gently heat to full redness with occasional stirring, being careful not to lose any of the material. Continue heating until all of the organic matter is burned away. Cool the crucible in a desiccator and weigh the residue. Calculate the loss as insoluble organic matter.

Tabulate as follows:

|                                    |       |
|------------------------------------|-------|
| Soluble matter . . . . .           | — %   |
| Insoluble organic matter . . . . . | — %   |
| Mineral matter . . . . .           | — %   |
| Total . . . . .                    | 100 % |

- How have you seen filtration employed outside of school?
- What is another name for the process?
- What kind of substances may be separated by filtration?
- Make a list of mixtures containing such substances.

**Application to Commercial Analysis of Tea.** The approximate value of tea may be determined by calculating the per cent of matter which is not soluble in hot water. The insoluble matter should not be in excess of 60 per cent. If over this amount, the presence of spent or exhausted leaves is indicated.

Place exactly 2 g. of the finely powdered tea in a balanced and folded filter paper. Extract with successive portions of boiling water until the filtrate runs clear. Dry the residue

at 100° C. Cool and reweigh. Calculate by difference in weight the per cent of water-soluble matter.

Test samples of tea of various prices, and answer the question, Does the price indicate the quality of tea based upon matter insoluble in boiling water?

What per cent of insoluble matter has the tea which is used at your home?

**To isolate Theine, the Alkaloid of Tea.** Extract a spoonful of high-grade tea in 50 cc. of boiling water. Filter the liquid and add 10 cc. of chloroform. Transfer the mixture to a separatory funnel, shake the contents vigorously for a minute, and allow the chloroform to settle. Draw it off into a clean, dry watch glass and allow it to evaporate at room temperature. Note the white, silky crystals and the pleasant odor of the theine.

Repeat the above experiment, substituting coffee for tea. The alkaloid of coffee is called what? It is identical with that of tea. Its chemical symbol is  $C_8H_{10}N_4O_2 + H_2O$ . What per cent of nitrogen does it contain?

**Application to a Domestic Analysis of Oysters.** The determination of the amount of water added to shucked oysters is important from the viewpoint of the consumer. The per cent of water that may be separated by means of a strainer or sieve should not exceed 15 per cent.<sup>1</sup> Weigh on a trip balance about a pint of the sample as purchased. Place in a flat-bottomed sieve and allow to drain into a weighed dish for twenty minutes, stirring gently from time to time. Determine the weight and per cent of the exterior liquor. Determine the weight and per cent of the drained oysters by difference.

<sup>1</sup> *Merck's Report*, July, 1910, p. 189.

## SECTION II

### ACIDS AND ALKALIS

**Detection in Everyday Compounds.** The instructor will show samples of acids in solid, liquid, and gaseous form, to do away with the impression that all acids are liquids.

Show samples of the common alkalis.

Discover the effect of acids and alkalis upon the various indicators — phenolphthalein, methyl orange, cochineal, carmine, and litmus.

Take home some litmus paper cut into small strips. Test different articles — foods at the table, substances in the kitchen, laundry, etc. Be sure that the substance tested, if a solid, is either dissolved in water or well moistened.

One pupil tested the following: cream of tartar, ashes, salt, milk, apple juice, borax, sugar, baking soda, washing soda, soap, vinegar, tea, kerosene, tooth powder, coffee, butter, stomach bitters.

Arrange your results in three columns, thus:

*Acid*

*Alkaline*

*Neutral*

Give the chemical names and symbols of as many of the above list as possible. What per cent of the foods eaten during the day were acid? alkaline? neutral?

Determine the reaction of the soil from your lawn or garden. What advantage is it to know this reaction?

Thoroughly moisten 20 to 50 g. of the soil and insert two pieces of litmus paper, one red, the other blue.

Allow them to remain undisturbed for an hour or even overnight.

What would you suggest as a suitable dressing for an acid or "sour" soil? for an alkaline one?

What plants have a distinctly acid reaction? Do you know of any fruits which are not acid? Find out the name of the acid which imparts the sour taste to the common sheep sorrel (*Rumex acetosella*), apples, oranges, grapes, rhubarb, etc. What acid is found in the membranous covering of nuts? Mention some of the alkalis which are found in nature.

## SECTION III

### ALKALIS IN TEXTILE ANALYSIS

**Determination of the Per Cent of Wool and Cotton in Fabrics.** Wool is soluble in a solution of NaOH or KOH. Cotton is insoluble in this reagent.

*Qualitative Work.* Place a small piece of the sample in a test tube or crucible. Cover with 20 per cent KOH

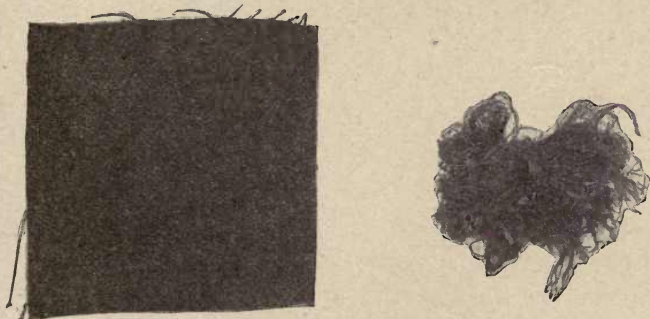


FIG. 1. Mixed goods heated with KOH, showing cotton residue

and boil for two or three minutes. If the sample entirely dissolves, what is the per cent of wool? Suppose it partially dissolves, what is indicated? Experiment with bits of cotton, wool, and mixed goods until you are familiar with the action of the caustic solution upon these substances.

**Caution.** Take great care that none of the hot alkali comes in contact with the flesh or clothing. If this does happen, apply dilute HCl at once.

*Quantitative Work.* If a portion of the cloth dissolves, cut a second sample about 8 cm. square. Determine the exact weight. Place in a beaker or large evaporating dish and cover with the caustic potash. Boil for three minutes, or until the wool is dissolved. Remove the cotton residue, taking care not to lose any detached threads. Rinse thoroughly. Add a drop of phenolphthalein and sufficient HCl to make slightly acid. Wash, dry at 100° C., and reweigh. Calculate the per cent of wool and cotton. Devise a method for testing silk.

## SECTION IV

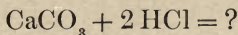
### ACIDS AND ALKALIS IN THE QUALITATIVE ANALYSIS OF SOILS

The appended list comprises the more important plant foods, and the majority of them can be easily detected in common soil.

|   |                               |
|---|-------------------------------|
| Water                                   | Sulfuric acid as sulfates     |
| Lime ( $\text{CaCO}_3$ )                | Hydrochloric acid as chlorids |
| Carbon dioxid                           | Nitric acid as nitrates       |
| Ferric oxid ( $\text{Fe}_2\text{O}_3$ ) | Magnesia ( $\text{MgO}$ )     |
| Soda ( $\text{Na}_2\text{O}$ )          | Sand ( $\text{SiO}_2$ )       |
| Potash ( $\text{K}_2\text{O}$ )         |                               |

**Calcium Carbonate.** Place 10 g. of the soil in a test tube and add 2 cc. of HCl. An effervescence indicates the presence of a carbonate. This effervescence may frequently be heard when the action is only faintly visible, by placing the ear near the mouth of the test tube.

Add enough water to make a thin paste and boil for about two minutes. Make alkaline with ammonia, and filter. Test the clear filtrate with an equal volume of  $(\text{NH}_4)_2\text{C}_2\text{O}_4$ . A cloudiness or flocculent precipitate, which forms on standing, indicates the presence of calcium.



**Ferric Oxid.** *First Test.* Boil 4 g. of the soil with 10 cc. of HCl (5 parts of acid and an equal volume of water). Filter and test half of the filtrate with ammonia added in

excess. A reddish-brown, flocculent precipitate assures the presence of iron in the sample of soil.

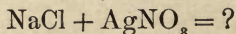
*Second Test.* To the other half of the filtrate add a few drops of potassium sulfo-cyanide (KCNS). A blood-red color is seen if ferric oxid is present in the original soil. Test further by adding a little  $\text{HgCl}_2$  to a cubic centimeter of the red liquid. The color should be destroyed. Write the reactions which take place in the first and second tests.

**Soda and Potash.** Boil 50 g. of the soil in an equal volume of water, stirring constantly. Allow the undissolved matter to settle and decant the clear liquid. Evaporate this, preferably over a water bath, to about 5 cc. Clean a platinum wire by heating until it gives no color to the blue Bunsen flame. Dip the wire into the concentrated filtrate and place immediately in the outer flame. A yellow coloration indicates soda. A lilac or violet color, best seen through a piece of cobalt-blue glass, indicates potash.

**Sulfuric Acid as Sulfates.** To a little  $\text{BaCl}_2$  solution add a few drops of the filtrate left from the soda and potash experiment. Boil. A precipitate insoluble in acids indicates the presence of sulfates. Write the reaction between sodium sulfate and barium chlorid.

**Hydrochloric Acid as Chlorids.** To two or three cubic centimeters of the soda filtrate add a few drops of  $\text{AgNO}_3$ . Churn the contents of the test tube. A white, curdy precipitate soluble in ammonia indicates the presence of chlorids in the soil.

Since other substances are likely to precipitate the silver solution, decant or filter off the ammoniacal solution and add an excess of  $\text{HCl}$ . If a second precipitate is seen or a decided milkiness is evident, the presence of chlorids is assured.





**Nitric Acid as Nitrates.** To one volume of the sodapotash filtrate add two volumes of strong  $\text{H}_2\text{SO}_4$  free from nitrates. Allow the mixture to cool. Incline the test tube and cautiously add a few drops of a concentrated solution of  $\text{FeSO}_4$ , so that the liquids will not mix. A brownish ring at the junction of the two solutions assures the presence of nitrates.

**Phosphoric Acid as Phosphates.** To 5 cc. of the sodapotash filtrate add a few drops of ammonium molybdate in  $\text{HNO}_3$ . Warm the evaporating dish gently. The presence of phosphates is indicated by a lemon-yellow color, or, if there is a considerable quantity present, by a yellow precipitate.

**Magnesia ( $\text{MgO}$ ).** Boil 25 g. of the soil in 50 cc. of water and 10 cc. of  $\text{HCl}$ . Filter and evaporate the filtrate to about 5 cc. Add a few drops of  $\text{NH}_4\text{Cl}$  and an equal volume of  $\text{NH}_4\text{OH}$ . If a precipitate forms, filter and test the filtrate with  $\text{HNa}_2\text{PO}_4$ .

A white crystalline precipitate of  $\text{MgNH}_4\text{PO}_4$ , soluble in acids, assures the presence of magnesium in the soil.

**Sand.** Wash 10 g. of the soil in a 250-cc. beaker with a small stream of water, stirring constantly until all the humus is washed out. Boil the residue in strong  $\text{HCl}$  for five minutes. Sand is the principal substance left.

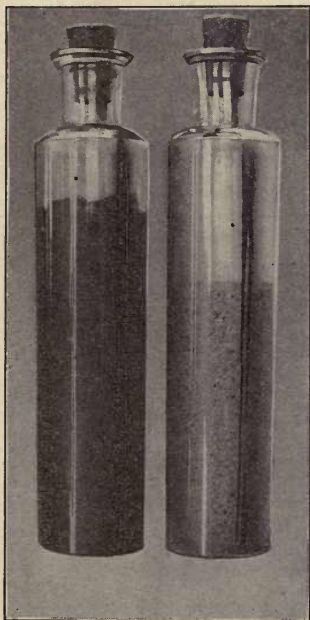


FIG. 2. Soil before and after boiling in strong  $\text{HCl}$ . The residue is sand

Examine with a magnifying glass. What material do you recognize? Can you suggest what some of the original rock might have been? How could you make this experiment quantitative?

Another important constituent of soils is organic nitrogen. Its detection and determination should be omitted until the pupil is familiar with the Gunning method for nitrogen, q.v.

## SECTION V

### DETECTION OF SOME OF THE COMPOUNDS PRESENT IN PLANTS

By far the greater part of the growing plant is carbon and water. How could you prove this?

This list comprises other compounds present in plants and vegetable matter.

|                          |            |
|--------------------------|------------|
| Organic nitrogen         | Phosphates |
| Chlorophyl               | Sulfates   |
| Starch                   | Chlorids   |
| Potash and soda          | Iron       |
| Manganese (infrequently) | Silica     |

**Organic Nitrogen.** Grind a small handful of grass or hay through a meat chopper. Mix with an equal weight of soda lime or with strong KOH, and heat gently in an Erlenmeyer flask, in the mouth of which is suspended a strip of moist red litmus paper. The ammonia given off will turn the test paper blue and prove the presence of organic nitrogen.

**Chlorophyl.** Chlorophyl is the green coloring matter common to growing plants.

Grind a handful of grass or green leaves, or scrape the green layer from any woody stem. Triturate in a mortar with enough alcohol to cover the mass. Decant or filter the green liquid. Allow the alcohol to evaporate spontaneously. The chlorophyl remains as an intensely green substance.

When extracted from suitable kinds of leaves, as spinach, parsley, etc., the chlorophyl is sometimes used for coloring confections, jellies, and beverages. Of what use is chlorophyl to plants?

**Starch.** Grind a few kernels of corn or any kind of grain; scrape a potato or bruise any kind of root. Boil with 10 cc. of water. Cool the contents of the tube and add a few drops of iodine. A distinct blue coloration is proof of the presence of starch.

**Potash and Soda. First Test.** Place a handful of dried grass in a white enameled pan or upon a porcelain tile. Light it with a blazing splint. When it has stopped burning collect the residue in a porcelain crucible. Repeat the experiment several times until the crucible is half full. Heat until the contents are thoroughly ashed. Sometimes the ash will be greenish, due to the presence of manganese, but more frequently it will be white or light brown. Care must be exercised lest the ash fuse to the crucible.

Wash the ash into a beaker and boil in 25 cc. of water. Allow the undissolved material to settle, and reserve. Examine the clear liquid directly for potash and soda by the flame test, as directed for the analysis of soils. If the results are not positive, evaporate 10 cc. of the liquid to dryness and repeat the test. Is the liquid acid or alkaline?

**Second Test.** Cut the bottom from a quart bottle or use a glass percolator. Loosely plug the neck of the percolator with absorbent cotton. Fill about two thirds full of sifted wood ashes, through which allow half a liter of warm water to pass. Collect the filtrate in a large evaporating dish or enameled pan. Evaporate to dryness, but do not char. The grayish-white substance is potash and soda, or the "lye" used in the early settlements for making soft soap. Dissolve

in a little water and treat with phenolphthalein, cochineal, or litmus. Apply the flame test.

**Phosphates, Sulfates, and Chlorids.** Separate the unused portion of the liquid obtained in the first or second test into three parts and apply the appropriate tests, as directed under soil analysis.

**Iron.** Boil the residue saved from the undissolved ash with a few cubic centimeters of HCl. Decant the liquid and test for iron with KCNS.

**Silica.** The residue from the iron test is mainly silica. Wash with water, allow to settle, decant the liquid, and rub the grayish material with the rounded end of a glass rod. A distinct scratching sound can usually be heard.

Tabulate those exercises in which you have used an acid or an alkali as a reagent. In which of these tests have you found an acid or an alkali?

## SECTION VI

### EXERCISES WITH STANDARD SOLUTIONS

**Titration and Standard Solutions.** The strength of many solutions may be determined with great accuracy by adding definite volumes of other solutions of known strength, which will react chemically with them. The process is known as titration, and is of great importance in chemical analysis.

The added solution is called a standard solution. It is made to contain an accurately determined weight of chemical substance in a definite volume.

A standard solution which contains 1 g. of replaceable hydrogen per liter is known as a normal solution. Standard solutions may be spoken of in terms of normal solutions, as  $1/2$ ,  $1/10$ ,  $1/50$ , etc., usually written  $N/2$ ,  $N/10$ ,  $N/50$ .

The amount of a given substance to be dissolved in distilled water and made up to a liter will vary directly as its molecular weight and inversely as the number of replaceable hydrogen atoms represented in its molecule. Thus a liter of normal  $HCl$  must contain 36.5 g. of acid, since its molecular weight is 36.5 and only one atom of replaceable hydrogen is present. A decinormal solution of this acid will contain 3.65 g. per liter, while a cubic centimeter will contain 0.00365 g.

A liter of normal  $H_2SO_4$  must contain 49 g. of acid, since its molecular weight is 98, and 2 atoms of replaceable hydrogen are present.  $N/10 H_2SO_4$  will contain 4.9 g. per liter.

A liter of normal oxalic acid ( $\text{H}_2\text{C}_2\text{O}_4 + 2\text{H}_2\text{O}$ ) must contain 63 g. of acid, since its molecular weight is 126 and it contains 2 atoms of replaceable hydrogen.

To prepare a liter of N/10 oxalic acid, weigh exactly 6.3 g. of acid of the highest purity, which has not lost its water of crystallization, and transfer to a liter measuring flask. Add about 500 cc. distilled water and agitate to dissolve the acid. This done, make up to the mark on the neck with more distilled water. Turn into a clean, dry bottle and mix thoroughly.

A cubic centimeter of any normal acid will exactly neutralize a cubic centimeter of any normal alkali. A liter of normal NaOH must contain 40 g. of the hydrate, since its molecular weight is 40. A liter of normal KOH must contain 56 g. of the salt because its molecular weight is 56.

That one may know when enough of a standard solution has been added to neutralize or to complete the reaction, it is customary to employ a third or neutral substance called an indicator. Litmus, phenolphthalein, cochineal, methyl orange, potassium chromate, etc. are often used.

Phenolphthalein is a good general indicator, but must not be employed when more than traces of  $\text{CO}_2$  are present. To prepare, dissolve 1 g. of the crystals in 100 cc. of 95 per cent alcohol. To use, add two or three drops of the alcoholic solution to the acid or to the alkaline solution under examination. In acid or in neutral solutions phenolphthalein is colorless, but the smallest excess of alkali turns it a vivid purple-red.

To prepare a liter of N/10 NaOH, weigh roughly 4 g. of the pure hydrate, transfer to a liter flask, and make up to the liter mark with distilled water. When the hydrate has dissolved, transfer to a large bottle and mix thoroughly.

The solution must now be standardized by the N/10 oxalic acid previously made.

By means of an accurate pipette transfer 10 cc. of the NaOH solution to a beaker. Add two or three drops of phenolphthalein. Set the beaker on a white tile or sheet of white paper and draw into it from a burette just enough N/10 oxalic acid so that the last drop destroys the lingering trace of pink in the alkali. Note carefully the amount of acid required.

Suppose 12 cc. are used; the alkali is too strong, for 1 cc. of the acid must neutralize 1 cc. of the alkali. Add 50 cc. of distilled water to the NaOH solution, mix well, and again titrate 10 cc. By observing the change made by the 50 cc. of water one may easily calculate the amount of water necessary to bring the alkaline solution to the exact tenth-normal strength.

Suppose only 8 cc. of the acid are required to neutralize the solution; the NaOH is too weak. Add a drop of saturated NaOH solution, mix well, and titrate a second 10-cc. portion. The requisite amount of alkali to be added may be calculated. Thus by adding water or alkali as required, an exact balance may be secured. Work for perfect standardization.<sup>1</sup>

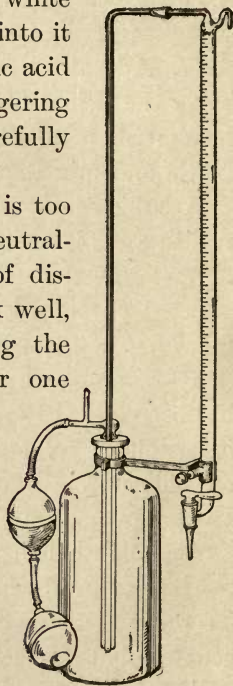


FIG. 3. A convenient type of burette

<sup>1</sup> For those wishing to go more into detail with volumetric solutions, Olsen's "Quantitative Analysis" (D. Van Nostrand Co.) will be helpful.



A cubic centimeter of N/10 NaOH is equal to

|  |           |
|--|-----------|
| Acetic acid ( $\text{HC}_2\text{H}_3\text{O}_2$ ) . . . . .                              | 0.0060 g. |
| Boric acid ( $\text{H}_3\text{BO}_3$ ) . . . . .   | 0.0062 g. |
| Citric acid ( $\text{H}_3\text{C}_6\text{H}_5\text{O}_7, \text{H}_2\text{O}$ ) . . . . . | .0070 g.  |
| Lactic acid ( $\text{HC}_3\text{H}_5\text{O}_3$ ) . . . . .                              | 0.0090 g. |
| Malic acid ( $\text{C}_4\text{H}_6\text{O}_5$ ) . . . . .                                | 0.0067 g. |
| Oxalic acid ( $\text{H}_2\text{C}_2\text{O}_4, 2 \text{H}_2\text{O}$ ) . . . . .         | 0.0063 g. |
| Sulfuric acid ( $\text{H}_2\text{SO}_4$ ) . . . . .                                      | 0.0049 g. |
| Tartaric acid ( $\text{H}_2\text{C}_4\text{H}_4\text{O}_6$ ) . . . . .                   | 0.0188 g. |

**Determine the Acidity of Oranges or Lemons calculated as Citric Acid.** Weigh or counterpoise a small, clean, dry evaporating dish. In this place 2 to 3 g. of orange juice free from pulp. If lemon juice is used, 1.5 to 2 g. is sufficient. Add two or three drops of phenolphthalein (*phenolphthalein, 1 g.; alcohol, 100 cc.*). Fill a burette to the zero mark with N/10 NaOH or take any other convenient mark as zero. Allow this standard solution to drop slowly into the fruit juice until the first permanent trace of pink appears in the well-mixed solution. If, after standing for a minute, the pink color disappears, add another drop of the alkali. The fruit juice has been titrated with N/10 alkali.

#### CALCULATION

1 cc. of N/10 NaOH is equivalent to 0.007 g. of citric acid.

|  |                |
|--|----------------|
| Suppose the weight of the orange juice and evaporating dish is . . . . . | 15.82 g.       |
| Suppose the weight of the evaporating dish is . . . . .                  | 13.30          |
| Weight of the orange juice is . . . . .                                  | <u>2.52 g.</u> |
| Suppose at the end of the reaction the burette reads . . . . .           | 13.80 cc.      |
| Suppose the zero point was . . . . .                                     | <u>10.50</u>   |
| Number of cubic centimeters of N/10 NaOH used . . . . .                  | <u>3.3 cc.</u> |

$3.3 \times .007 = .0231$  g. of citric acid in the 2.52 g. of juice. Therefore citric acid is present to the extent of 0.916 per cent.

Test the acidity of commercial lime and lemon juices. The U.S.P. requires about 7 per cent of citric acid.

**Determine the Purity of Cream of Tartar.** Place exactly half a gram of the sample in a clean beaker and dissolve in boiling water. Add the phenolphthalein solution as in the preceding experiment and titrate with decinormal NaOH. After the first pink color appears, heat nearly to boiling. If the color disappears, add another drop of the alkali. Half a gram of pure cream of tartar requires approximately 26.6 cc. (26.595) of N/10 NaOH to neutralize it.

If your balance is sufficiently accurate, weigh exactly 0.188 g. Dissolve in hot water and titrate, using phenolphthalein. If pure, it will require 10 cc. of the N/10 alkali.

**Determine the Purity of Baking Soda.** Every cubic centimeter of N/10 baking soda must contain .0084 g. of  $\text{HNaCO}_3$ . Why?

Titrate exactly half a gram of the sample with N/10 HCl, using methyl orange as an indicator.

How many cubic centimeters were required? Suppose the sample were absolutely pure, how many cubic centimeters of the N/10 acid would be required to neutralize it?

What is the per cent of purity? Write the reaction between HCl and  $\text{HNaCO}_3$ .

**Determine the Per Cent of Lactic Acid in Milk.** Fresh milk should not contain over .2 per cent of acid. Cream for the best butter should contain from .5 to .65 per cent.

Into a clean white evaporating dish or teacup place 9 cc. of milk or cream, being sure to rinse the graduate into the cup. Titrate with N/10 NaOH, using phenolphthalein as an indicator. Stir the contents of the cup frequently with a glass rod. The rod must not be taken from the dish lest some of the contents be lost. Neither must alkali

from the burette spatter upon it. The first permanent tint of pink in the milk indicates the end of the operation. The color should remain pink after one minute.

The number of cubic centimeters of the standard alkali used, indicates directly in tenths of one per cent the amount of acid present. Thus:

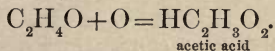
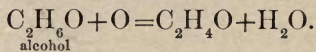
6 cc. of the alkali = .6 per cent of acid.

9.3 cc. of alkali = .93 per cent of acid.

**An Exercise in testing Vinegar.** "Vinegar is formed by the action of an organism found in the 'mother' of vinegar upon weak alcoholic solutions.

This organism serves in some way to carry the oxygen of the air to the alcohol. The sour taste is 'largely due to acetic acid' (*Remsen*).

The following equations illustrate the process of so-called acetic fermentation:



The physical and chemical examination of vinegar

is full of interest. Examine a few drops under a low-power microscope and see if you can find any of the so-called "vinegar eels." They are best seen after allowing a large test tube of vinegar to remain undisturbed for several hours. The eels are usually near the surface of the liquid.

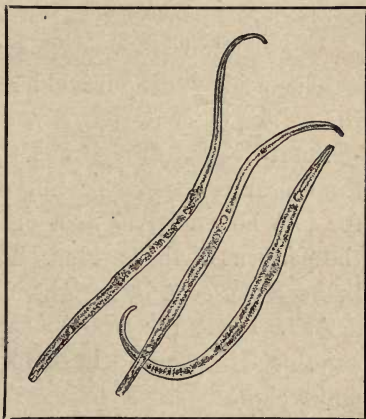


FIG. 4. "Vinegar eels." (Magnified)  
(Wilson)

**Total Acidity.** Introduce exactly 6 cc. of the sample into a clean beaker and set upon a white paper or tile in a good light. Titrate with N/10 NaOH, using phenolphthalein as an indicator.

The number of cubic centimeters of the decinormal alkali used, divided by 10, represents the per cent of acetic acid.

#### CALCULATION

Suppose 34.3 cc. of the standard alkali are required to neutralize 6 cc. of the vinegar; then  $\frac{34.3}{10} = 3.43$  per cent of acetic acid.

*Proof.* 1 cc. of N/10 NaOH neutralizes 0.006 g. of acetic acid.

$34.3 \times .006 = 0.2053$  g. of acetic acid.

Therefore 6 g. (or 6 cc.) of vinegar contain 3.43 per cent of acetic acid.

The acidity of pure cider vinegar should not be less than 4.5 per cent.

**Total Solids.** Weigh exactly 10 g. of the vinegar into a tared crucible or evaporating dish and evaporate to dryness for two hours. Cool in a desiccator, weigh, and calculate the per cent of the residue. The total solids should not fall below 2 per cent by weight if the sample is cider vinegar.

**Ash.** Place the vessel containing the total solids on a suitable triangle and burn to ash at a low red heat. Cool and weigh.

The ash should not be less than 0.25 per cent. Its reaction to litmus should be distinctly alkaline. If it is not, it contains some mineral acid which must be determined. Gather some of the ash on the loop of a clean platinum wire and apply the flame test. View through the cobalt-blue glass if necessary. What base is present?

Add a few drops of 10 per cent HCl. What radical is evident? In the ash of pure cider vinegar potassium carbonate is prominent.

**Ashby's Test for Mineral Acids.** Prepare a solution of log-wood by dissolving 0.5 g. of the extract in 100 cc. of boiling water. Place one drop of this solution on the bottom of an evaporating dish and dry over a water bath. To the dried residue add a drop of vinegar and dry again. Pure vinegar gives a yellow stain. Free mineral acids color the residue red.

To be sure of the Ashby reaction, it is well to add a little  $H_2SO_4$  to vinegar of known purity and compare the result with the residue under examination.

**To detect Sulfuric Acid in the Presence of Natural Sulfates of the Vinegar.** Evaporate 100 cc. of the sample to one tenth of its volume, and when cold add 50 cc. of alcohol. Sulfuric acid remains in solution while the natural sulfates are precipitated. Dilute the solution and precipitate the  $H_2SO_4$  with  $BaCl_2$ . Write the reaction.

**Detection of Caramel.** Caramel, or burned sugar, is sometimes used to make the vinegar seem stronger than it really is, or to make it resemble cider vinegar. Shake 5 cc. of the sample with twice its volume of amyl alcohol. If caramel is present, the supernatant layer will be wholly or partially decolorized. The under layer will be a deep brown, depending upon the amount of caramel present.

**Detection of Coal-Tar Dye.** Use the double-dyeing process of Sostegni and Carpentieri, q.v.

**To distinguish Cider Vinegar from Other Vinegars.** *Lythgoe's Method.* Prepare lead subacetate solution by dissolving 180 g. of lead acetate in half a liter of water. Add 110 g. of  $PbO$  and make up to 1000 g. with water. Agitate often and filter.

To 25 cc. of the vinegar add 2.5 cc. of the subacetate solution. Shake the contents of the tube. The precipitate should be copious and settle out in a few minutes.

For comparative results, centrifuge in a graduated tube and read the volume of the precipitate. The amount present, in part at least, indicates the value of the vinegar.

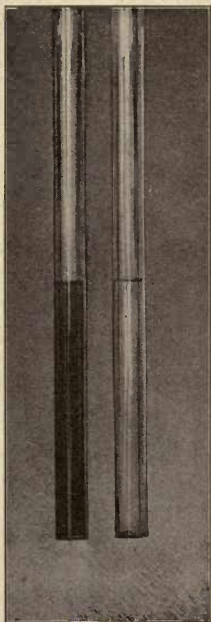


FIG. 5. True and artificial vinegar treated with lead subacetate

The artificial vinegar, on the right, shows little or no effect

Unless a precipitate is formed the sample is not cider vinegar.

**Determination of the Approximate Acidity or Alkalinity of Soils.** Boil exactly 10 g. of thoroughly dried soil in 30 cc. of water. Filter and wash the residue several times with small portions of hot water. Add the washings to the filtrate with two or three drops of phenolphthalein. If the reaction is alkaline, titrate with N/10 oxalic acid until the pink color is destroyed by addition of the last drop.

If the reaction is acid, titrate with N/10 NaOH until the first tinge of pink appears.

In either case express the result as the number of cubic centimeters of the N/10 solution required to neutralize the filtrate.

**Approximation of the Purity of Cocoa.** The purity of cocoa can, in part at least, be determined by calculating the per cent of its ash, which in pure samples seldom exceeds 5.5 per cent; and under ordinary conditions the ash from each gram of the sample will require not more than 3.7 cc. of N/10 oxalic acid to neutralize it. If possible, test samples from your home.

Burn 2 g. of the sample to a carbon-free ash at the lowest possible heat. Cool the crucible and calculate the per cent of ash. Boil the contents of the crucible in 50 cc. of water, being sure to rinse it well and save the washings. Titrate with N/10 oxalic acid, using phenolphthalein as an indicator.

**Analysis of Soap. Insoluble Matter.** Dissolve 5 g. of the sample in 75 cc. of water, heating if necessary. If there are more than mere traces of residue, filter on a tared filter paper, wash with hot water until the filtrate is neutral, dry at 105° C., and calculate the per cent.

Test various "hand" and scouring soaps for insoluble matter.

Alkali may exist in soap in at least three forms, — free, combined, and as alkaline carbonates, borates, etc.

**Detection of Free Alkali.** Treat the freshly cut surface of the soap with a few drops of the alcoholic solution of phenolphthalein. If no red color appears, it may be assumed that free alkali is absent. Take great care that no water comes in contact with the soap, otherwise the results may be misleading.

When testing washing powders for free alkali, dissolve a small quantity in alcohol and add the phenolphthalein.

**Combined Alkali.** If free alkali, alkaline carbonates, borates, etc. are absent, dissolve a gram of the soap in 20 cc. of hot water. Treat with phenolphthalein. A red color is immediately seen. Why?

Titrate with N/10 oxalic acid. Allow the acid to drop slowly from the burette until the last drop added destroys the pink color. Shake or stir the solution frequently during the operation and be sure to waste none. If after a minute the pink color reappears, add another drop of the acid.

Free alkali may also be estimated by dissolving it from 1 g. of the soap by means of alcohol. Filter, wash with alcohol, and titrate as for combined alkali. Calculate as NaOH.

#### CALCULATION FOR FREE OR COMBINED ALKALI

1 cc. of N/10 oxalic acid neutralizes 1 cc. of N/10 NaOH, or 0.004 g. of NaOH. Suppose the number of cubic centimeters of acid required is 4.75; then the per cent of alkali present is  $4.75 \times .004 = .019$ , or 1.9 per cent.

Write the reaction between oxalic acid and sodium hydrate.

**Total Alkali.** If the soap is free from sand and other mineral matters, burn 2 g. to ash in a porcelain or quartz crucible. Cool and wash the contents into 50 cc. of water. Boil and titrate with N/10 oxalic acid, using two drops of methyl orange as an indicator. (*Dissolve 1 g. of methyl orange in 1 liter of water.*)

Express the result as the acid number; that is, the number of cubic centimeters of decinormal acid necessary to neutralize the alkali in 1 g. of the soap.

**Alkaline Carbonates.** Place 5 g. of the soap in an Erlenmeyer flask and add 20 cc. of alcohol. Set a funnel in the neck of the flask to act as a reflux condenser, and heat on a water bath for ten minutes. Alkaline carbonates will be in the residue. Place a bit of this residue on a clean platinum wire and apply the flame test for sodium and potassium (see under Soil Analysis). Filter the alcohol and dissolve the residue in warm water. Add a few drops of dilute HCl. A marked effervescence indicates the presence of carbonates; whether  $\text{Na}_2\text{CO}_3$  (washing soda) or  $\text{K}_2\text{CO}_3$  (potash) will be indicated by the flame test.



**Borates.** Place 5 cc. of turmeric tincture in a watch glass. Add a few drops of the soap dissolved in water, and acidify slightly with dilute HCl. Evaporate to dryness over a water bath. The presence of borates is indicated by the pronounced reddening of the dried residue.

Record the results of your analysis in this form :

| SAMPLE | PER CENT<br>INSOLUBLE<br>MATTER | PER CENT<br>FREE ALKALI | PER CENT<br>COMBINED<br>ALKALI | CARBONATES | BORATES |
|--------|---------------------------------|-------------------------|--------------------------------|------------|---------|
|        |                                 |                         |                                |            |         |

## SECTION VII

### SANITARY ANALYSIS OF WATER

The importance of wholesome water cannot be overestimated. Do the barns, the sinks, the outbuildings, contaminate the supply? Does the water dissolve poisonous metals from the pipes or other sources? Is the water hard or soft? All these questions we shall be able to answer.

Clear, sparkling, odorless water may be totally unfit for domestic purposes, while a suspicious-looking or peculiar-smelling sample may be quite harmless. A simple chemical analysis is often of great value, except in the detection of specific disease germs, when an intelligent bacteriological examination is necessary. The analysis in this case, however, may throw much light upon the source of the contamination.

Keep carefully tabulated results of your analyses. You can then see at a glance how the water varies from the normal.

**Sediment.** Allow a test tube or conical glass full of the water to stand overnight, or centrifuge 10 cc. in a pointed tube. If any sediment falls, decant the liquid and examine the deposit under a microscope. This deposit may be divided into two classes, harmless and suspicious matter: harmless matter, *sand, clay, algæ, diatoms*; suspicious matter, *hair, epithelial scales, bits of wool and cotton, muscle fibers, etc.*

Filter the remainder of the original sample before making further tests.

**Color.** Fill a clean test tube, the longer the better, with the water, or use a Nessler tube. Stand it on a white paper or tile, facing a good light. Cover the back of the tube, except an inch at the bottom, with a piece of white paper. On looking down through the tube the water should be perfectly transparent, or show only a faint bluish tinge. Pollution is indicated by tints of green, yellow, or brown.

**Odor.** Warm about 250 cc. of the water to  $38^{\circ}\text{C}$ . in a corked flask. Shake, remove the stopper, and smell the contents. Pure water is free from odor. The odor may be classified as earthy, vegetable, alkaline, putrid, etc.

A putrid odor indicates decomposing animal or vegetable matter. If much polluted by fresh sewage, the odor of urine is not infrequent. One should note that many waters unfit to drink have no odor. A positive odor teaches volumes; a negative result is of little value.

**Total Solids.** These consist for the most part of  $\text{CaCO}_3$ ,  $\text{MgSO}_4$ ,  $\text{CaSO}_4$  with their chlorids and nitrates,  $\text{NaCl}$ ,  $\text{SiO}_2$ , and organic matter.

Evaporate 70 cc. of the water to dryness in a thin, counterpoised evaporating dish over a water or steam bath. Dry, cool in a desiccator, and weigh as milligrams. Every milligram of solids per 70 cc. represents one grain per gallon

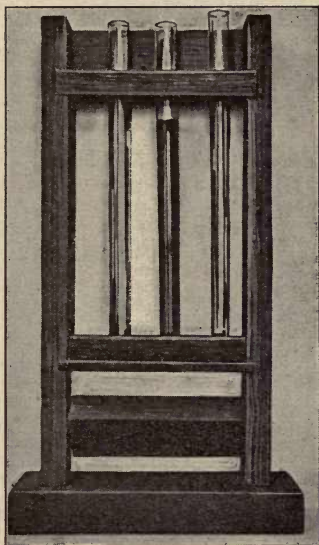


FIG. 6. A convenient rack for Nessler tubes

The movable mirror shows at an angle

in the original sample. Total solids in good water may be as high as 30 grains per gallon.

Save the residue and examine for phosphates. The residue from pure water is almost white. Iron gives a yellow or coppery luster to the sides of the dish.

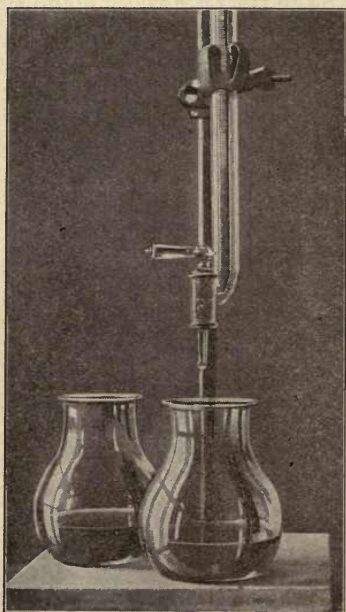


FIG. 7. Apparatus for the determination of chlorine in water

Control flask on the left

tion. Do not add more than a drop at a time. Continue the titration with frequent agitation of the contents until the water shows the first tinge of red. The first trace is best seen by looking at the titrated sample through the colored water in the control flask.

The number of cubic centimeters of the silver solution required to produce the red tinge equals the number of

Evaporate about 50 cc. of the sample in a porcelain evaporating dish and heat gently at first. Charring denotes the presence of organic matter.

#### Determination of Chlorides

Measure 50 cc. of the water with a pipette into each of two small flasks or beakers. Add three or four drops of potassium chromate solution, 10 per cent, as an indicator, coloring the contents of each flask exactly alike. Place both flasks on a white tile in a good light. Titrate the water in one of the flasks with a standard silver solu-

parts of chlorin in 100,000 parts of water. If the water contains more than five parts of chlorin per 100,000, contamination from human urine or sink drains is to be suspected, unless the water is taken near the seacoast or from some locality where the normal sodium chlorid content is above this amount. This information can usually be obtained from any state board of health.

Write the reaction between silver nitrate and potassium chromate.

The standard silver solution is prepared by dissolving 2.3944 g. of pure  $\text{AgNO}_3$  in a liter of distilled water. Keep this solution in a yellow or black bottle away from the light.

**Detection of Ammonia.** To ~~25~~<sup>50</sup> cc. of the water in a Nessler tube, tall test tube, or foot tube, add ~~25~~ cc. of Nessler's reagent and note the color. A faint yellow tinge only should be visible. A deeper color or turbidity indicates animal contamination. P. J. C.

Compare the treated water with an equal volume of the untreated sample in a similar tube.

The experiment may be made quantitative by comparing the color of the sample with different Nesslerized samples of distilled water which contain known quantities of  $\text{NH}_4\text{Cl}$ .

All natural waters contain a trace of ammonia, but the amount present should not be sufficient to cause more than a slight coloration with the Nessler solution. An exception must be made in the case of rain water, which, although relatively pure, contains a considerable amount of ammonia dissolved from the atmosphere. Rain water, however, shows practically no nitrates or chlorin.

*Nessler's Reagent.* Dissolve 62.5 g. of KI in 250 cc. of water. Reserve about 10 cc. of this solution. Run into the remainder a cold, saturated solution of  $\text{HgCl}_2$  until a permanent precipitate forms.

Redissolve this precipitate by means of the reserve KI solution. Very cautiously add more of the mercuric chlorid solution until a slight precipitate remains after agitation. Add 150 g. of KOH in water and make up to a liter.

Allow the precipitate to settle and decant or siphon off the clear liquid. This solution improves with age.

**Detection of Nitrites.** Into a Nessler tube place a drop of HCl, 2 cc. of sulfanilic acid, an equal volume of naphthylamine hydrochlorid, and 50 cc. of the water under examination. If a red color is produced immediately or within twenty minutes, the presence of nitrites is assured.

As a rule nitrites are never found in good water.

*Sulfanilic Solution.* Dissolve 0.8 g. of the acid in 100 cc. of pure water, heating if necessary.

*Naphthylamine Hydrochlorid Solution.* Dissolve 0.8 g. of the salt in 100 cc. of hot water to which 1 cc. of HCl has been added. Filter through bone black or add bone black to the solution, and decant as needed. Keep from the light.

**Detection of Nitrates.** Evaporate 100 cc. of the sample to dryness in a white evaporating dish over a water or steam bath. Treat with 1 cc. of phenol-sulfonic acid, stirring thoroughly. Add 10 cc. of distilled water and half as much  $\text{NH}_4\text{OH}$ .

In the presence of nitrates the characteristic yellow color of the ammonia salt of nitrophenol-sulfonic acid is formed.

Nitrates are present in almost all natural terrestrial waters.

**Detection of Phosphates.** Evaporate 70 cc. of the sample to dryness or use the residue from the determination of total solids. Add a few drops of ammonia molybdate in nitric acid and warm gently. There should be only a slight lemon-yellow coloration. A decided yellow coloration indicates animal pollution. The degree of intensity is sometimes recorded as traces, heavy traces, and very heavy traces.

*Ammonium Molybdate Solution.* Dissolve 15 g. of ammonium molybdate in 100 cc. of water, with the addition of a little ammonia if necessary. If there is pronounced turbidity, filter the solution and pour with constant stirring into a mixture of 50 cc. nitric acid and an equal volume of water. Allow the solution to stand in a warm place for several days at a temperature of about 80° F. and decant the clear liquid.

*Determination of ~~Absorbed~~ Oxygen.* This test gives reliable information concerning the amount of organic contamination, but does not distinguish between that of animal and vegetable origin.

If more than one grain per gallon is absorbed, the water is probably polluted.

*Preliminary Test.* Fill two test tubes half full, one with distilled water, the other with the sample. To each add a drop of strong  $H_2SO_4$  and sufficient  $KMnO_4$  in distilled water to color each a very light purple, as nearly alike as possible. Boil the contents of each tube. What is the action of organic matter on  $KMnO_4$ ?

*Regular Test.* Prepare a standard solution of  $KMnO_4$ , 0.395 g. per liter, and keep in a clean well-stoppered bottle.

Place exactly 70 cc. of the water to be examined in a clean flask and add ten drops of 10 per cent  $H_2SO_4$ . Warm gently and add the standard solution from a burette, drop by drop, shaking the flask gently after the addition of each drop. As soon as the faintest tinge of pink appears, warm the flask again and notice whether the color is permanent. If not, add another drop. The first permanent tinge of pink indicates the end of the operation.

Limit the test to fifteen minutes. Use an ordinary Bohemian flask.

When 70 cc. of water is thus titrated, each cubic centimeter of the permanganate solution represents 0.1 of a grain per gallon. Good water absorbs less than a grain per gallon.

**Metallic Compounds. Lead.** Evaporate 100 cc. of the sample to 20 cc. and add a few drops of  $K_2Cr_2O_7$ . A precipitate of chrome yellow assures the presence of lead.

**Iron and Copper.** Boil 2 g. of stearic acid for five minutes in 30 cc. of the suspected water. Set aside, and when cool compare its color with a sample of the acid that has been boiled for the same length of time in distilled water. Examine before a white background. Iron salts impart a yellow color. If traces of copper are present, the acid will be colored a bluish green, which can be seen even before the acid has solidified.

**Hardness.** Titrate 100 cc. of the water with N/10 HCl, using methyl orange or erythrosin as an indicator. The number of cubic centimeters of the decinormal acid used, multiplied by 50, represents the number of parts of  $CaCO_3$  per million parts of water. Calculate the results to parts per 100,000. Water is considered "hard" if it contains over five parts of  $CaCO_3$  per 100,000.



## SECTION VIII

### EXAMINATION OF BAKING POWDER

All baking powders leave a more or less insoluble residue in the food which they are used to leaven. What salts does one take into his system when he eats cake or biscuit made with cream of tartar, with alum, or with phosphate baking powder? The following reactions are of interest:

| KIND OF BAKING POWDER     | REACTIONS   |
|---------------------------|---|
| Cream of tartar . . . . . | $\text{HKC}_4\text{H}_4\text{O}_6 + \text{HNaCO}_3 = \text{KNaC}_4\text{H}_4\text{O}_6 + \text{CO}_2 + \text{H}_2\text{O}$ <div style="display: flex; justify-content: space-around; font-size: small;"> <span>Potassium bitartrate</span> <span>Rochelle salts</span> </div>                       |
| Alum . . . . .            | $\text{K}_2\text{Al}_2(\text{SO}_4)_4 + 6 \text{HNaCO}_3 = \text{Al}_2(\text{OH})_6 + 3 \text{Na}_2\text{SO}_4$ <div style="display: flex; justify-content: space-around; font-size: small;"> <span>Burnt alum</span> <span>Glauber's salts</span> </div> $+ \text{K}_2\text{SO}_4 + 6 \text{CO}_2$ |
| Phosphate . . . . .       | $\text{H}_4\text{Ca}(\text{PO}_4)_2 + 2 \text{HNaCO}_3 = \text{HCaPO}_4 + \text{HNaPO}_4$ <div style="display: flex; justify-content: space-around; font-size: small;"> <span>Calcium acid phosphate</span> </div> $+ 2 \text{CO}_2 + 2 \text{H}_2\text{O}$   |

**Determination of Carbon Dioxid.** The value of a baking powder, in part at least, depends upon the amount of available  $\text{CO}_2$  it contains. To determine this, two separate tests are necessary.

**Total Carbon Dioxid.** This determination consists of liberating the  $\text{CO}_2$  from a weighed amount of the sample, passing the gas through caustic potash, and ascertaining the increase in weight. Into an Erlenmeyer flask of about 150 cc. capacity weigh exactly 2 g. of baking powder. Fit the flask with a dropping funnel and a delivery tube inclined upward at an angle of about  $20^\circ$ . To the free

end of the delivery tube attach a drying tube filled with granulated  $\text{CaCl}_2$  previously saturated with  $\text{CO}_2$ .

Fill a set of Liebig potash bulbs two thirds full of a solution of 1 part  $\text{KOH}$  and 2 parts water. Determine the exact weight of the prepared bulbs by suspending from

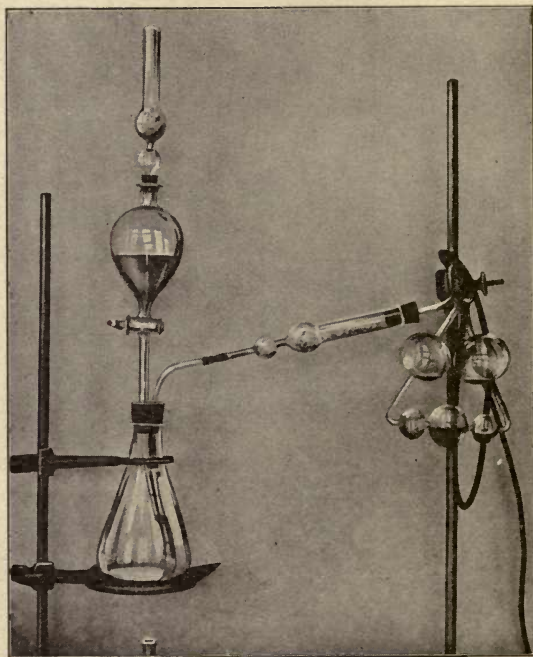


FIG. 8. Apparatus for the determination of carbon dioxide

one arm of the balance. This done, connect the inlet of the bulbs with the free end of the drying tube, and the outer end with an aspirator.

Fill the dropping funnel half full of  $\text{HCl}$  (sp. gr. 1.1).

Place a small drying tube filled with soda lime in the mouth of the funnel, to prevent any  $\text{CO}_2$  from the air

being drawn into the potash bulbs when the aspirator is in operation.

Open the funnel slightly and allow the acid to drop slowly upon the sample. Adjust the aspirator so that the gas will be drawn through the potash bulbs at the rate of about two bubbles per second. When nearly all the acid has been added, heat the contents of the generating flask to boiling, until the water begins to condense in the delivery tube.

Aspirate until the potash bulbs are cool; then disconnect and reweigh. The increased weight is due to total carbon dioxid.

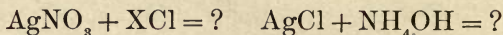
Write the reaction between HCl and baking soda. Is the absorption of  $\text{CO}_2$  by KOH due to physical or chemical change?

**Residual Carbon Dioxid.** Thoroughly clean the apparatus used in the determination of total carbon dioxid. Introduce 2 g. of the sample into the generating flask and add 20 cc. of cold water. Allow it to stand for twenty minutes. Then set the flask into a tin can surrounded by boiling water for the same length of time. Drive off the last traces of gas in the pasty mixture by boiling for one minute. Aspirate until the air is thoroughly changed. At this point in the experiment connect the apparatus and perform the work exactly as for the determination of total carbon dioxid. The increase of weight in the potash bulbs is due to the residual carbon dioxid.

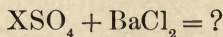
**Available Carbon Dioxid.** From the per cent of total carbon dioxid subtract the per cent of residual carbon dioxid.

The average per cent of available carbon dioxid found in the three principal kinds of baking powder is as follows: cream of tartar, 12.58; phosphate, 12.86; alum, 8.10 (*Bulletin No. 13, United States Bureau of Chemistry*).

**Tests for Radicals found in Baking Powder. Chlorids.** Shake 2 to 4 g. of the sample with 25 cc. of cold water. Filter. This constitutes a cold-water extract. To 5 cc. of this extract add a few drops of 10 per cent  $\text{AgNO}_3$  solution. If a precipitate forms, agitate and allow it to settle. If it is insoluble in hot water, but dissolves in ammonia, the presence of chlorids is indicated.



**Sulfates.** Treat a few cubic centimeters of the cold-water extract with barium chlorid solution. A precipitate insoluble in acids and in water assures the presence of sulfates.



*Royal*  
**Tartrates.** Dissolve a crystal of silver nitrate in 5 cc. of water and add two drops of ammonia. Add 1 cc. of the cold-water extract and heat gently. If tartrates are present, the silver will be deposited as a beautiful silver mirror on the interior of the tube.

**Carbonates.** To a gram or so of the dry baking powder in a test tube add a few drops of dilute  $\text{HCl}$ . Hold a glass rod wet with limewater so that the escaping gas will come in contact with it. If the limewater becomes milky, the presence of a carbonate is assured.

Write the reaction between hydrochloric acid and bicarbonate of soda. Write the reaction between carbon dioxide and limewater.

**Phosphates.** Add a few drops of the cold-water extract to a cubic centimeter of ammonium molybdate  $(\text{NH}_4)_2\text{MoO}_4$  in  $\text{HNO}_3$ . Heat gently. A lemon-yellow precipitate indicates the presence of phosphates.

Phosphoric acid is an important constituent of the body. Its presence can be easily demonstrated by experiment.

Burn a piece of bone in a clear fire until the residue is perfectly white. Powder from 2 to 3 g. of this and dissolve in HCl. Dilute the solution about one half and add an excess of ammonia. A white, gelatinous precipitate of calcium and magnesium phosphate forms. Filter, and to the filtrate add ammonium oxalate. The characteristic precipitate of  $\text{CaC}_2\text{O}_4$  is evident.

What other radical was present in the bone?

**Tests for Bases found in Baking Powder. Calcium.** To a test tube half full of the extract add a few drops of ammonium oxalate  $(\text{NH}_4)_2\text{C}_2\text{O}_4$ . A precipitate insoluble in acetic acid, but soluble in hydrochloric acid, shows the presence of calcium.

Write the reaction between ammonium oxalate and calcium acid phosphate.

**Ammonia.** Boil 50 cc. of the extract with 25 cc. of 10 per cent NaOH. Test the steam with red litmus paper. Avoid touching the neck of the flask with the paper. If present, the liberated ammonia will turn the test paper blue.

Write the reaction between ammonium carbonate and sodium hydrate.

**Aluminium.** (*Thirty-first Report of the Massachusetts State Board of Health.*) Burn to ash about 2 g. of the baking powder in a crucible. Extract with boiling water, and filter. To the filtrate add sufficient  $\text{NH}_4\text{Cl}$  to give a distinct odor of ammonia. A flocculent precipitate indicates the presence of aluminium. The lower grades of baking powder often contain salts of aluminium or of ammonium.

**Detection of Alum in Pastry as well as in Baking Powder.** Make a tincture of logwood by digesting 5 g. of the well-powdered chips in 100 cc. of alcohol. Prepare a saturated solution of  $(\text{NH}_4)_2\text{CO}_3$ .

Phosphate - Phosphate

Rub 5 g. of the baking powder, cake, cooky, or biscuit in a mortar with 10 cc. of water. Add 2 cc. of the logwood mixture and an equal volume of the ammonium carbonate solution.

If alum is present, the color changes to lavender or blue, and does not disappear on boiling. If alum is not present, the color varies from red to pink.

## SECTION IX

### ANALYSIS OF MILK

More disease and fraud enter the home through the milk supply than through any other article of food. Chemistry is of untold benefit in protecting people from these evils.

What is the quality and condition of the milk which you are using at home ?

**Fat.** Examine a drop of milk under a half-inch objective and note the collection of various-sized fat globules. Describe their appearance and arrangement. The fat of milk is

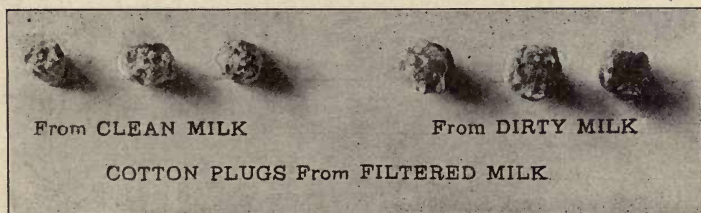


FIG. 9

one of its most variable constituents, and the determination of the amount present is of importance.

**Determination of the Per Cent of Fat by the Babcock Method.** This method consists in adding strong sulfuric acid to the milk to dissolve all of the solids except the fat, which is afterwards separated by means of a centrifugal machine.

Measure exactly 17.6 cc. of the milk into a Babcock bottle by means of a milk pipette. Add exactly 17.5 cc. of  $H_2SO_4$ ,

specific gravity 1.83 at 60° F., inclining the bottle so that the acid will run in slowly and wash all adhering milk from the neck.

Shake with a rotary motion so as to thoroughly mix the acid and milk. Avoid getting curds into the neck of the bottle.

If the work has been done properly, the mixture will be a dark brown color and very hot. Place directly into a centrifuge, arranging the bottle so that the rotating head

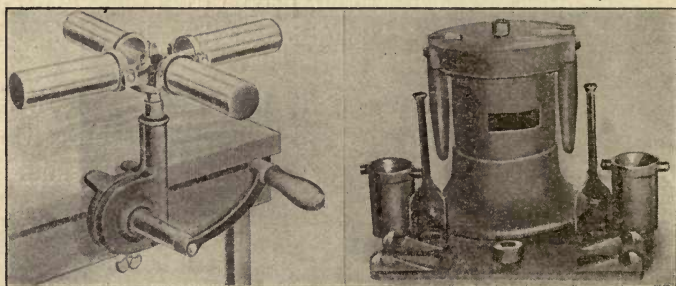


FIG. 10. Hand and electric centrifuges for milk analysis or for sedimentation

will balance properly. If the machine vibrates badly, the balance is not correct, and it must be adjusted.

Centrifuge for five minutes; then set the bottle into a pan of hot water and add sufficient hot water to bring the fat up to the neck of the bottle.

Centrifuge for two minutes and add hot water sufficient to bring the fat opposite the graduated scale.

Centrifuge for one minute and take the reading in tenths of 1 per cent directly from the scale.

A pair of small dividers is useful for determining the length of the fat column. This determined, place one leg of



the dividers upon zero and take the reading from the opposite leg. It is customary to take the distance from the bottom of the fat column to the top of its meniscus as the true length.

The fat in milk varies from 2.2 per cent to 9.0 per cent. The United States standard is 3.25 per cent. What is the standard in your state?

**Determination of Acidity.** Test both with red and blue litmus paper. Perfectly fresh cow's milk is generally alkaline. It is sometimes amphoteric, that is, it exhibits the phenomenon of reacting alkaline with red litmus and acid with blue. The acidity increases as the milk sugar is converted into lactic acid.

For quantitative determination of lactic acid, see page 20.

**Specific Gravity.** Determine with a lactometer, specific-gravity flask, or Westphal balance. Milk of good standard quality should have a specific gravity of 1.027–1.033 at 60° F.

Milk whose specific gravity varies from these limits is of a suspicious character.

**Total Solids, by Evaporation.** Heat 2 g. of milk to a constant weight in a counterpoised dish over a water bath.



FIG. 11. Babcock bottles for milk and cream with dividers to facilitate reading

The residue of milk must not be heated over  $100^{\circ}\text{C}$ ., as it will decompose and lose weight almost indefinitely. If properly heated, the dried residue will be almost white.

#### Total Solids, by Richmond's Slide Rule.

This method is by far the more convenient, and accurate enough for all practical purposes.

Take the temperature of the milk and the lactometer reading. Set this reading on the slide opposite the observed temperature. The corrected reading will be found opposite 60 on the scale. Call this reading  $A$ .

Place the arrow at the right-hand end of the slide, opposite the per cent of fat found by the Babcock method. Find  $A$  on the opposite edge of the slide. It will coincide with the total solids in the milk.

The results obtained by these two methods should agree closely.

Total solids should not be less than 12.50 per cent.

#### Tests for Foreign Matter, Dirt, Hair, etc.

Some of the most disagreeable as well as dangerous kinds of foreign matter which contaminate the milk supply enter through the carelessness of the producer.

Construct a percolator by cutting the bottom from a pint bottle, or use an ordinary glass percolator employed by a druggist. Insert a plug of clean absorbent cotton in



FIG. 12. Richmond's slide rule for the determination of total solids in milk

the neck from the inside, and allow a half pint or more of the well-shaken milk to slowly filter through. Remove the plug carefully and examine (see p. 41).

Insoluble matter, if present, can easily be seen. Wash the clean end of the plug gently with cold water. Dry and mount on cardboard with an appropriate inscription.

**Tests for Adulterants: Artificial Colors.** Coloring matter is sometimes added to milk to give it a rich, creamy appearance. The practice, if not absolutely injurious, is at least reprehensible. Why?

The two most commonly employed colors are annatto and coal-tar dye.

**Detection of Annatto.** Shake about 5 cc. of the milk with twice its volume of ether in a large test tube. When the liquids have separated, pour off the ether extract. Evaporate on a water bath. Make the residue alkaline with NaOH and pour on a small wet filter paper. The annatto will be absorbed by the pores of the paper.

Wash off the fat gently with slightly warmed water. Annatto will give a decided orange tone, which turns to pink when treated with a few drops of stannous chlorid.

**Detection of Coal-Tar Dye.** To 10 cc. of the milk add an equal volume of HCl and mix thoroughly. A pink coloration indicates the presence of azo orange.

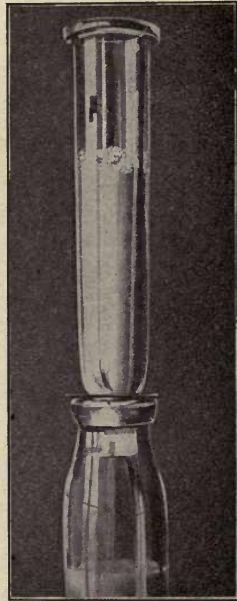


FIG. 13. Percolating milk, for dirt and other foreign matter

**Tests for Adulterants: Preservatives.** The preservatives most commonly employed to keep milk sweet for a longer time than nature intended are formaldehyde, compounds of boron (*borax and boric acid*), sodium bicarbonate, and calcium succrate.

**Detection of Formaldehyde (HCHO).** This is one of the most poisonous of preservatives found in foods, and when so used cannot be too strongly condemned.

*Leach's Casein Test. First Part.* To 10 cc. of pure milk add an equal volume of hydrochloric acid, containing about 1 per cent of  $\text{Fe}_2\text{Cl}_6$ . Use an evaporating dish or casserole and heat slowly, stirring or shaking the contents constantly to break up the curd. When nearly but not quite boiling, remove the heat and note the color of the treated milk when hot and when cold.

*Second Part.* To 50 cc. of water add 1 cc. of HCHO and mix the liquids well. Add two drops of this dilute formaldehyde to 10 cc. of pure milk and perform the work as directed under the first part.

How is the presence of formaldehyde indicated? How much was in the milk, assuming that the cubic centimeter added to the water contained 40 per cent formaldehyde?

The work under the first and second parts is designed to make the pupil familiar with the appearance of milk which contains and which does not contain formaldehyde, when the samples are treated with hydrochloric acid containing a small percentage of ferric chlorid.

To test any given sample of milk, proceed as directed under the first part, and if the results are similar to those obtained under the second part, the presence of formaldehyde is indicated. Both this and the following test are exceedingly delicate and the results are thoroughly reliable.

*Detection of HCHO by means of Hehner's Ring.* To 25 volumes of  $H_2SO_4$  add 1 volume of ferric chlorid solution. Place 4 cc. of this reagent in a large test tube and carefully add 5 cc. of the suspected milk, inclining the tube so that the milk shall rest upon the surface of the acid. In the presence of formaldehyde a violet ring is seen at the contact of the two liquids.

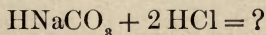
**Detection of Boron Compounds.** *First Part.* Place 5 cc. of pure milk in a watch glass and acidulate slightly with 10 per cent HCl. Add five drops of turmeric tincture and evaporate to dryness over a water bath. What is the appearance of pure milk thus treated?

*Second Part.* To 5 cc. of the milk add a drop of boric acid or borax dissolved in a little water. Transfer to a watch glass and proceed as before. Take care that the mixture does not char. How is boron indicated? The reaction is said to be sensitive to 1 part of boric acid in 25,000 parts of milk. The red color is proportionate to the amount of boron present.

Test unknown samples of milk, performing the work as outlined under the first part. If the sample of milk is very rich, or if cream is under examination, dilute with two or three volumes of water.

*Turmeric Tincture, U.S.P.* Digest any convenient amount of ground turmeric root in small quantities of water, discarding the liquids. Digest the dried residue with six times its weight of alcohol, and filter.

**Detection of Bicarbonate of Soda.** The ash of pure milk shows no effervescence with HCl. Burn 10 cc. of milk to a white ash in a porcelain or quartz crucible over a low flame. Treat the ash with a drop of 10 per cent HCl. An effervescence indicates bicarbonate of sodium.



**Detection of Calcium Succrate.** This substance is used as a thickener as well as a preservative, and is more frequently found in cream than in milk. It may be readily detected by means of the Baier and Neumann test which follows:

*First Part, the Sugar.* To 25 cc. of the milk or cream add 10 cc. of a 5 per cent solution of uranium acetate. Shake and allow to stand for five minutes.

Filter; if the filtrate is not clear, pour it through again. To 10 cc. of the clear filtrate (if the sample is cream, use the total filtrate) add 2 cc. of a cold saturated solution of ammonium molybdate freshly prepared and 8 cc. of dilute HCl (1 part of 25 per cent HCl and 8 parts of water). Agitate the mixture well and place the small flask containing it in a water bath at 80° C. for ten minutes.

If the sample is pure, the solution will be a peculiar green, resembling nickel sulfate solution; but if sugar is present, it will be a Prussian blue color.

*Second Part, the Calcium.* Evaporate 25 cc. of the milk or cream to dryness and burn to ash in a muffle. Dissolve the ash in 20 cc. of N/10  $H_2SO_4$ . Boil to expel the  $CO_2$  and titrate back with N/10 NaOH, using phenolphthalein as the indicator.

Express the results as cubic centimeters of N/10 acid required to neutralize 100 g. of milk or cream. Suppose that the ash was dissolved in 20 cc. of N/10 acid and that 14 cc. N/10 NaOH neutralized the excess. It is evident that 6 cc. of the acid was required to neutralize the alkalinity of the ash.

**Detection of Gelatin in Milk, Cream, and Ice Cream.** This substance is sometimes used in cream, and more frequently in ice cream, either as a thickener or to make the material stand transportation better. Gelatin is readily detected by the Stokes test.

**Stokes's Test.** To 10 cc. of the sample add an equal volume of acid nitrate of mercury solution and 20 cc. of cold water. Shake the mixture vigorously and allow it to stand for five minutes. Filter; if gelatin is present, the filtrate will be opalescent. Confirm by treating the filtrate with 1 cc. of a saturated aqueous solution of picric acid. The gelatin will be precipitated as a yellow solid, more or less flocculent in appearance.

**Acid Nitrate of Mercury Solution.** Dissolve any convenient weight of mercury in twice its weight of concentrated  $\text{HNO}_3$  and dilute this solution to 25 times its bulk with water.

**Detection of Milk adulterated by Skimming and by Watering.** In order to do this work intelligently one must become familiar with the principal factors employed in calculations of this kind. These factors are:

Total solids: all the constituents of milk except water.

Fat.

Solids not fat: obtained by subtracting the fat content from the total solids.

Proteins: the nitrogenous part of the milk.

Ash: the mineral constituents.

Milk sugar and lactose.

The following table, devised by H. C. Lythgoe, is believed to show the limits between which normal milk varies:

|                          | Extreme limits<br>(per cent) | Usual limits<br>(per cent) | Herd milk<br>(per cent) |
|--------------------------|------------------------------|----------------------------|-------------------------|
| Total solids . . . . .   | 10.0 — 17.0                  | 10.5 — 16.0                | 11.8 — 15.0             |
| Fat . . . . .            | 2.2 — 9.0                    | 2.8 — 7.0                  | 3.2 — 6.0               |
| Proteins . . . . .       | 2.1 — 8.5                    | 2.5 — 4.5                  | 2.5 — 4.0               |
| Ash . . . . .            | 0.6 — 0.9                    | 0.7 — 0.8                  | 0.7 — 0.8               |
| Solids not fat . . . . . | 7.5 — 11.0                   | 7.7 — 10.0                 | 8.0 — 9.5               |
| Milk sugar . . . . .     | 4.0 — 6.0                    | 4.2 — 5.5                  | 4.3 — 5.3               |

A relation has been found to exist between the fat and proteins of milk. If the fat is given, the proteins may be approximately calculated by means of Van Slyke's formula:

$$0.4(F. - 3) + 2.8 = P.$$

Suppose the fat found in a certain sample of milk is 3.50 per cent. According to the formula, the proteins are 3.0 per cent.

A relation also exists between the total solids and proteins, which is expressed by Olsen as

$$T.S. - T.S./1.34 = P.$$

Suppose the solids in the sample mentioned above were 12.50 per cent. The proteins, by Olsen's formula, are 3.18 per cent. If the milk is pure, the per cent of proteins calculated by the two formulas will agree closely with a variation of approximately 0.2 per cent or less. If the milk is adulterated with water or by skimming, the results will not agree, the difference increasing with the amount of adulteration.

*Illustrations.* A sample of milk known to be watered contained 3.4 per cent fat and 10.41 per cent of total solids.

|   |       |
|---|-------|
| Proteins calculated from the fat . . . . .    | 2.96% |
| Proteins calculated from the solids . . . . . | 2.65% |
| Difference . . . . .                          | .31%  |

A sample of milk known to be skimmed contained 2 per cent fat and 11.18 per cent of total solids.

|   |       |
|---|-------|
| Proteins calculated from the fat . . . . .    | 2.40% |
| Proteins calculated from the solids . . . . . | 2.84% |
| Difference . . . . .                          | .44%  |

According to Lythgoe we may use these formulas in the indirect calculation of milk sugar, if we assume the average ash of milk to be 0.7 per cent.

$$T.S. - (F. + [0.4(F. - 3) + 2.8] + 0.7) = \text{Milk sugar.}$$

$$T.S. - (F. + [T.S. - T.S./1.34] + 0.7) = \text{Milk sugar.}$$



**Detection of Skimmed Milk.** If the sample has been skimmed, the calculated proteins will exceed the fat, and the calculated milk sugar will be too high, exceeding 4.8 per cent, which is approximately the average milk sugar, according to the table.

If the fat is less than 2.2 per cent and the solids not fat are above 8.5 per cent, the milk has probably been skimmed.

**Detection of Added Water, Copper Sulfate Method.** Since there is no chemical test to distinguish between added water and the water naturally present in milk, it is customary to precipitate the fat and proteins by means of acetic acid, copper sulfate, or by spontaneous souring. This leaves the fat and protein in the curd, the milk sugar and ash in the whey.

Dissolve 72.5 grains of pure crystallized  $\text{CuSO}_4$  in a little water and dilute to 1 liter. Adjust this to have a specific gravity of 1.0443 at 20° C. compared with water at 4° C.

To 1 part of copper solution add 4 parts of sweet milk, shake thoroughly, and filter.

If the specific gravity of the clear filtrate is less than 1.0245 at 20° C., compared with water at 4° C., added water is indicated.

Dry 5 cc. of the copper serum to a constant weight over a water bath. Determine the weight of the 5 cc. from the specific gravity of the serum and calculate the total solids. If the total solids are below 5.28 per cent, added water is indicated.

## SECTION X

### EXAMINATION OF ICE CREAM, CHEESE, AND CONDENSED MILK

**Determination of the Fat in Ice Cream.** Tare a Babcock cream bottle and add 10 g. of the well-mixed sample together with 5 or 6 cc. of water. Mix thoroughly and add just enough sulfuric acid to turn the contents dark brown. Avoid an excess of acid, as the mixture will char badly. Proceed with the regular Babcock test. Multiply the scale reading by 18 and divide by 10, to find the per cent of fat.

The per cent of fat in ordinary cream is also found by this method.

**Starch.** Boil a small quantity of the sample in a test tube with 10 cc. of water. Cool, and add a few drops of iodine tincture. The well-known blue color will be evident if the sample contains starch.

If starch is present, place a drop of the sample on a slide and examine with the microscope to see if you can determine the kind.

**Gelatin.** Apply Stokes's test.

**Fat in Cheese.** Weigh 6 g. of the sample in a tared beaker. Add 10 cc. of boiling water and a few drops of ammonia. Stir gently until a smooth emulsion is formed. Transfer the contents to a Babcock cream bottle, cool slightly, and add 17.6 cc. of  $H_2SO_4$ , first rinsing out the beaker with the acid. Proceed with the regular Babcock test.

The fat reading on the scale multiplied by 18 and divided by the weight of the sample (6 g.) gives the per cent of fat in the cheese.

How can you distinguish a full-cream cheese from a skimmed-milk cheese?

**Fat in Condensed Milk (Unsweetened).** Weigh 6 g. of the thoroughly mixed sample into a tared Babcock milk bottle and add enough water to make the volume up to about 17.6 cc. Mix, and add sufficient  $H_2SO_4$  to produce the deep brown color required by the Babcock method. About 14 cc. of acid will be needed. Proceed with the regular Babcock test.

Multiply the fat reading by 3, to find the correct per cent of fat in the sample.

**Number of Times Condensed and Fat in the Original Milk.** The average ash of pure milk is .70 per cent. Burn from 2 to 3 g. of the sample in a crucible and calculate the per cent of ash. This per cent divided by .7 gives approximately the number of times the milk has been condensed.

Divide the fat found by the Babcock method by the number of times condensed, to find the fat in the original sample.

## SECTION XI

### DISTILLATION EXPERIMENTS

Prepare a solution of 2 g. of NaCl in 50 cc. of water. Taste. Add a few cloves and a little sand, and color with red ink or dye. Pour into a retort or distilling flask. Adjust a suitable condenser and distil off about one half.

Examine both residue and distillate. What substances are present in the distillate that were present in the original mixture? What kinds of substances are separated by distillation? What physical principle is involved? What practical applications of distillation do you know of? What is fractional distillation?

**Extraction of Essential Oils.** The odor and taste of many vegetable substances is due, in large measure, to the presence of a volatile compound known as essential oil.

What is the difference between fixed and essential oils?

Extract the essential oil from wintergreen leaves, sweet birch (*Betula lentula*), cloves, cinnamon, nutmeg, peppermint, bay leaves, orange or lemon rind.

To obtain the greatest amount of oil from any of these substances, grind through a meat chopper, add water to make an extremely thin paste, and distil. At first the distillate will be of a milky appearance, due to the suspended oil. Set aside until the liquid clears.

A second method for the extraction of essential oils, which gives even better results than the first and removes the liability of breaking the retort through the heavy

material sticking to the bottom, is to place the finely ground mass in a dry flask fitted with a two-hole stopper and connected with a condenser. Steam from a generating flask is blown in through a long bent glass delivery tube, which extends nearly to the bottom of the flask.

What is the use of essential oils? What is the meaning of "essence," "extract," and "tincture"? How may essential oils be extracted except by distillation? (See *Harpers' Monthly for November, 1906.*)

**Experiments with Alcohol.** Few substances are of greater industrial value than alcohol. Make a list of the uses of alcohol, either ethyl ( $C_2H_5OH$ ) or methyl ( $CH_3OH$ ), or both. What is denatured alcohol?

Usually from 5 to 8 per cent of grain alcohol is sufficient to preserve the extractive principles of medicine. How do you account for such high amounts as 28 to 44 per cent sometimes present?

Make a medicine tester. Cut a piece of quarter-inch glass tubing 8 in. long. Smooth the ends. Push one end through a one-hole rubber stopper. To the free end fit an ordinary Welsbach burner and mantle.

Place two tablespoonfuls of the liquid under examination in a Bohemian flask and insert the stopper and tube. Heat the flask gently, and if alcohol is present in amounts of over 12 per cent, it will cause the mantle to glow brightly when ignited.

To get the best effect, inclose the mantle in a glass or mica chimney. Some brands of "bitters," "tonics," "cures," "specifics," etc. contain enough alcohol to keep up the incandescence for five or six minutes.

Instead of the Bohemian flask one may substitute a copper can fitted with a small screw stopper. Into the top of

the can solder a piece of gas pipe about 8 in. long and fit with a burner. Be sure that all connections are well made.

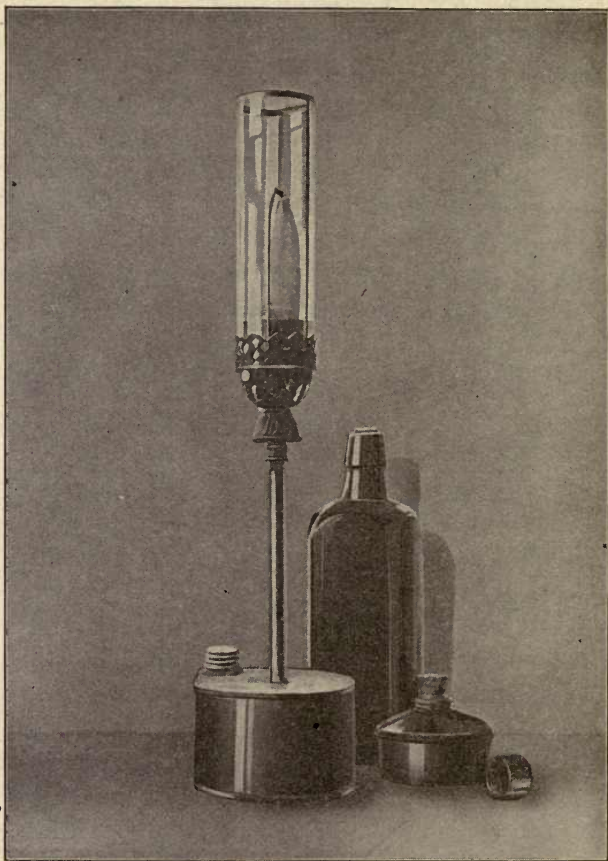


FIG. 14. Testing apparatus to demonstrate the presence of alcohol in medicine and beverages

**Preparation of Alcohol ( $C_2H_5OH$ ).** Dissolve 30 g. of cane sugar in 75 cc. of water, add a drop of  $HCl$ , and boil one

minute. If grape sugar or 20 cc. of molasses is used instead of cane sugar, the boiling and acid may be omitted.

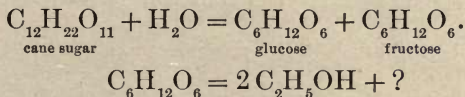
Dissolve half a cake of yeast in the same volume of water not above 50° C. Cool the sugar solution to about the same temperature and mix with the yeast. Allow to remain in a warm place for two or three days, and observe from time to time any visible changes.

Decant the liquid into a retort and distil off about one half. Wash the retort, pour in the distillate, and distil off 10 cc. Test the second distillate as follows:

Determine its odor and taste. In it test the solubility of a bit of camphor or the oil from a piece of orange or lemon peel. Burn a little in an evaporating dish. To equal quantities of the distillate and acetic acid add a few drops of  $H_2SO_4$ , taking care that the contents of the tube do not spatter in your face. Heat gently. Notice the ethereal, fruitlike odor of ethyl acetate, or acetic ether, a substance much used in the preparation of artificial flavoring compounds.

If the experiment has been conducted properly, the fermented liquid will contain about 4.5 per cent of alcohol by volume.

Cane sugar does not ferment. Treated properly with acid the reaction probably takes place as follows:



The instructor or one of the pupils should prepare a flask of the sugar-yeast mixture. Fit a delivery tube so that its free end dips below the surface of a few cubic centimeters of limewater contained in a small, narrow-necked

bottle. The presence of  $\text{CO}_2$ , given off during the process of fermentation, is thus easily demonstrated.<sup>1</sup>

**Determination of the Per Cent of Alcohol by Distillation.** The chemical and physical characteristics of alcohol are so apparent that a qualitative analysis is seldom necessary. It is highly important, however, to know how much of this substance enters into the composition of various medicines and articles intended for internal use. To comply with the Food and Drugs Act, the amount of alcohol must be plainly stated upon the label. How does your analysis agree with the statement?

Introduce exactly 100 cc. of sweet cider, beer, wine, medicine, root-beer extracts, or any of the various "tonics" and "blood purifiers," into a retort and distil off about one half, being sure that the free end of the condenser dips deeply into the receiving flask. Make up the distillate to exactly 100 cc. by adding distilled water. Determine the specific gravity of the thoroughly mixed distillate, and from this determine the per cent of alcohol by volume by means of the tables (pp. 66-75).

Repeat the experiment by weighing exactly 100 g. of the sample in a tared flask or beaker. Transfer to a retort and distil as before.

Make the distillate up to 100 g. with distilled water. Take the specific gravity. From this determine, by means

<sup>1</sup> The student will find helpful references to alcohol in the following publications:

*Popular Science Monthly*: "History of Alcohol," Vol. LI, p. 231; "Physiology of Alcohol," Vol. L, p. 796; "Natural Production of Alcohol," Vol. XIX, p. 238; "Discovery and Distillation of Alcohol," Vol. XLIII, p. 85; "Use of Alcohol in Medicine," Vol. XXXVIII, p. 86; "Vinous Superstitions," Vol. XXIV, p. 234; "Pigs as Wine Bibbers," Vol. XXIV, p. 426.

*Farmers' Bulletin No. 268*, United States Department of Agriculture.



of the tables, the per cent of alcohol by weight. Discover any mathematical sequence in the alcohol tables (pp. 66-75).

*Suggestions.* Always carefully rinse the vessel containing the measured amount of the sample into the retort, using about half its volume of water.

Determine the temperature of the distillate at which you take the specific gravity and correct to 60° F. or 15° C. For all temperatures above these standards subtract .00080 for each Centigrade degree or .00044 for each Fahrenheit degree. For all temperatures below these standards the correction is additive.

If, for this work, pupils are encouraged to bring samples from home, the results will often be

a revelation not only to themselves but also to their parents.

**Determination of the Per Cent of Alcohol by Difference in Specific Gravity.** This method, suggested by Leach, gives approximate results. Use the preceding method whenever possible.

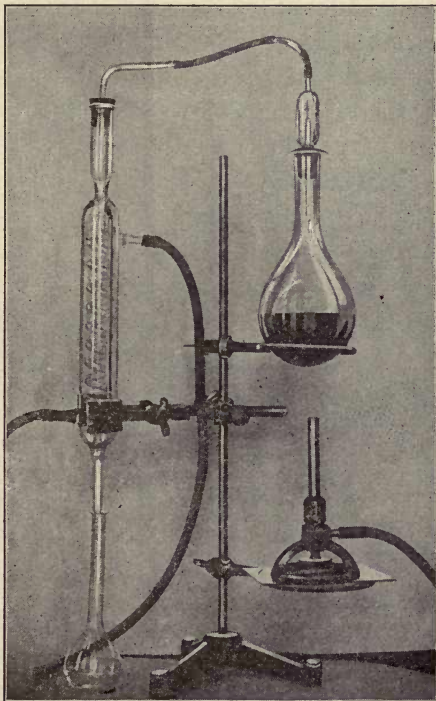


FIG. 15. Apparatus for determining the per cent of alcohol

Determine the specific gravity of the original sample. Evaporate 50 cc. over a water bath to one fourth its bulk. Make up to its original volume with distilled water. Determine the specific gravity of this dealcoholized portion. Add 1 to the original specific gravity and subtract the second. The difference corresponds to the alcohol in the original sample.

Consult the tables (pp. 66-75) as before and read the per cent of alcohol by volume.

**Methyl or Wood Alcohol ( $\text{CH}_3\text{OH}$ ).** It is difficult to find a more dangerous liquid used to cheapen food products and medicinal preparations than methyl alcohol. It is responsible for many cases of death and blindness. It apparently makes little difference whether it is taken internally in medicines and beverages, rubbed on the skin as a liniment, or its vapor inhaled; death, severe illness, or blindness may result.<sup>1</sup>

In more instances than one this poison has been found, in its deodorized form, in Jamaica ginger, lemon extract, peppermint and cinnamon, "hot drops," liniments, bitters, toilet waters, bay rum, witch hazel, spirits of camphor, paregoric, whisky.

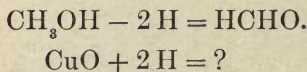
**Detection of Methyl Alcohol.** Except where a refractometer is used, the presence of wood alcohol is proved indirectly, usually by oxidizing it into formaldehyde.

Cut a piece of No. 14 copper wire 90 cm. long. At a point 20 cm. from one end wind the wire neatly around a pencil into a close spiral until about 20 cm. from the opposite end. Push the first end through the coil and twist both together to form a handle.

<sup>1</sup> For instances of methyl alcohol poisoning, see *Journal of the American Medical Association*, October 1-29, 1904.

Prepare a solution of 1 part  $\text{CH}_3\text{OH}$  and 6 parts water. Mix, and pour 10 cc. into a 5-in. test tube whose inside diameter is a little greater than the diameter of the spiral. Stand the tube in a bottle of cold water, to cool the contents during the oxidizing process.

Heat the coil to a dull red in the upper Bunsen flame and plunge immediately into the dilute alcohol. Withdraw in a second and dip into water. Repeat the operation from three to six times. This treatment will change all or a part of the alcohol into formaldehyde.



What reaction takes place when the copper spiral is heated in the upper Bunsen flame?

Decant the oxidized liquid and divide into two portions. Add one portion to a little milk and apply the tests for formaldehyde.

To the other portion add 1 drop of 1 per cent resorcin solution and mix well. Pour this mixture carefully down the side of a wide test tube which contains a half inch of concentrated  $\text{H}_2\text{SO}_4$ . A red ring (not brown) will form between the two liquids. If much formaldehyde is present, a precipitate may be seen. This is known as Hehner's resorcin test.

Test witch hazel, lemon extract, cheap vanilla, etc. by oxidizing 10 cc. as indicated. In the case of vanilla it is advisable to distil off about 10 cc. and oxidize the distillate.

**Caution.** One must always be sure that the original sample does not contain formaldehyde before testing for methyl alcohol, otherwise the results might be misleading.

If there is a question, add a few drops of the original sample to 5 cc. of milk and apply the Hehner test.

All aldehydes may be removed by distilling 50 cc. of the sample with 10 g. of sodium bisulfite and allowing the mixture to stand for two hours. Distil a second time, and make

distinctly alkaline with NaOH. Oxidize this distillate and test for methyl alcohol (*Report Massachusetts State Board of Health, 1906, p. 401*).

**Use of Alcohol in the Preparation of Vanilla Extract.** The preparation of flavoring extracts from pure and high-grade materials is a valuable experience for students of chemistry. The work can be done in the laboratory or at home.

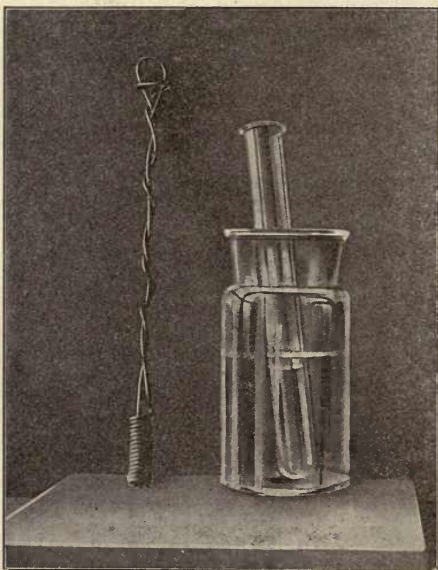


FIG. 16. Copper spiral and water-cooled test tube for the oxidation of methyl alcohol into formaldehyde

Vanilla extract is the flavoring extract prepared from vanilla beans, with or without sugar or glycerin, and contains in 100 cc. the soluble matters from not less than 10 g. of the beans (*U. S. standard*).

*Preparation (United States Pharmacopœia).* Cut 100 g. of vanilla beans in a meat chopper, which must be clean and free from foreign odors. Weigh 200 g. of coarse

granulated sugar; mix 650 cc. of alcohol with 350 cc. of water.

Macerate the vanilla in 500 cc. of this dilute alcohol for twelve hours; then drain off the liquid and set it aside. Transfer the vanilla to a mortar and beat it with the sugar. Pack the mass into a percolator, the neck of which has been plugged with a piece of absorbent cotton from within, and pour on the reserved dilute alcohol. When this has disappeared from the surface, gradually pour on the menstruum and continue percolation until 1000 cc. have been obtained. Repercolate several times.

Vanilla extract improves with age. If the alcoholic mixture remains on the beans for several months, the flavor of the extract will be improved.

**To distinguish between True and Artificial Vanilla Extract.** Many of the so-called vanilla extracts contain little or no vanilla, but are composed of a dilute alcoholic solution of vanillin, or coumarin, prepared from coal tar. Sometimes true coumarin, the aromatic principle of the tonka or "snuff" bean, is added to make the flavor more pronounced and lasting. Such extracts are often colored with caramel or with coal-tar dye.

Some experience is necessary to distinguish between true and artificial extracts by a sense of smell or taste alone, but there are several chemical tests which will definitely settle the matter.

*By the Lead Acetate Precipitate.* To 40 cc. of the sample add an equal volume of normal lead acetate solution prepared by dissolving 189.5 g. of  $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 + 3 \text{H}_2\text{O}$  in water and diluting to 1 liter.

The absence of a precipitate is conclusive evidence that the extract is artificial. Pure vanilla thus treated yields a

heavy precipitate which should settle in a few minutes, leaving a clear, partially decolorized liquid (*Leach*).

*By Examination of the Resins Present.* Evaporate the alcohol from 50 cc. of the extract over a water bath and make up to the original volume with water. If an alkali (usually  $K_2CO_3$ ) has not been used in the manufacture of the extract, the resins will appear as a flocculent red to brown residue. Acidify with acetic acid, allow to stand for a few minutes, and collect the resins on a filter paper. Wash the residue twice with slightly warmed water.

Tear off a portion of the filter paper with the resins attached, and place it in a few cubic centimeters of dilute KOH. If the vanilla is pure, this resin will dissolve, making a deep red solution.

Dissolve the rest of the resin in alcohol and divide the solution into three parts:

(a) Add a few drops of ferric chlorid. There will be no particular change if the vanilla is pure.

(b) Add a few drops of dilute HCl. If the vanilla is pure, there will be no particular color change.

(c) Add a few drops of lead subacetate solution. If the vanilla is pure, the precipitate will be so bulky as to almost solidify. Its filtrate will be almost colorless (*Bulletin No. 107, revised*).

#### Use of Alcohol in the Preparation of Lemon Extract.

Lemon extract is the flavoring extract prepared from the oil of lemon or from lemon peel, or both, and contains not less than 5 per cent by volume of lemon oil (*U. S. standard*).

An extract of fair quality, but which does not contain the legal amount of lemon oil, may be prepared by grating the yellow rind from six lemons and macerating in 40 per cent alcohol for a few days. It can then be filtered and used.

A legal extract can be prepared by dissolving 1 oz. of lemon oil in 19 oz. of alcohol. This extract can then be colored, if desired, by the addition of some of the bright yellow solution prepared from the lemon peel.

**Approximate Test for Strength and Purity.** To 50 cc. of cold water add 2 cc. of the lemon extract. The oil will be thrown out of solution, giving the top of the water column a decided milky appearance. The depth of the milkiness, in a measure, enables one to judge of the strength of the extract. Absence of the milky color is conclusive proof that the extract is artificial. If it is of a decided yellow color, test for artificial color as directed under Coal-Tar Dye.

**Per Cent of Lemon Oil. *Mitchell's Test.*** Place 20 cc. of the extract in a Babcock milk bottle. Add 1 cc. dilute HCl (1 to 1) and about 28 cc. of warm water at 60° C. Mix and allow the bottle to stand in warm water at the same temperature for five minutes. Centrifuge for five minutes. Add warm water to bring the oil into the graduated neck. Centrifuge for two minutes more and stand the bottle in water at 60° C. up to the top of the oil column for a few minutes, and read the per cent of oil by volume.

If more than 2 per cent of oil is present, add 0.4 per cent to correct for the oil retained in solution. If between 1 and 2 per cent, add 0.3 per cent for correction.

TABLE FOR DETERMINATION OF ALCOHOL PERCENTAGES<sup>1</sup>

BY SQUIBB, DRINKWATER, AND GILPIN

| SPECIFIC GRAVITY AT 60° F. | ALCOHOL            |                    | SPECIFIC GRAVITY AT 60° F. | ALCOHOL            |                    | SPECIFIC GRAVITY AT 60° F. | ALCOHOL            |                    |
|----------------------------|--------------------|--------------------|----------------------------|--------------------|--------------------|----------------------------|--------------------|--------------------|
|                            | Per cent by volume | Per cent by weight |                            | Per cent by volume | Per cent by weight |                            | Per cent by volume | Per cent by weight |
| 1.00000                    | 0.00               | 0.00               | 0.99775                    | 1.50               | 1.19               | 0.99557                    | 3.00               | 2.39               |
| 0.99992                    | 0.05               | 0.04               | .99768                     | 1.55               | 1.23               | .99550                     | 3.05               | 2.43               |
| .99984                     | 0.10               | 0.08               | .99760                     | 1.60               | 1.27               | .99543                     | 3.10               | 2.47               |
| .99976                     | 0.15               | 0.12               | .99753                     | 1.65               | 1.31               | .99536                     | 3.15               | 2.51               |
| .99968                     | 0.20               | 0.16               | .99745                     | 1.70               | 1.35               | .99529                     | 3.20               | 2.55               |
| .99961                     | 0.25               | 0.20               | .99738                     | 1.75               | 1.39               | .99522                     | 3.25               | 2.59               |
| .99953                     | 0.30               | 0.24               | .99731                     | 1.80               | 1.43               | .99515                     | 3.30               | 2.64               |
| .99945                     | 0.35               | 0.28               | .99723                     | 1.85               | 1.47               | .99508                     | 3.35               | 2.68               |
| .99937                     | 0.40               | 0.32               | .99716                     | 1.90               | 1.51               | .99501                     | 3.40               | 2.72               |
| .99930                     | 0.45               | 0.36               | .99708                     | 1.95               | 1.55               | .99494                     | 3.45               | 2.76               |
| .99923                     | 0.50               | 0.40               | .99701                     | 2.00               | 1.59               | .99487                     | 3.50               | 2.80               |
| .99915                     | 0.55               | 0.44               | .99694                     | 2.05               | 1.63               | .99480                     | 3.55               | 2.84               |
| .99907                     | 0.60               | 0.48               | .99687                     | 2.10               | 1.67               | .99473                     | 3.60               | 2.88               |
| .99900                     | 0.65               | 0.52               | .99679                     | 2.15               | 1.71               | .99466                     | 3.65               | 2.92               |
| .99892                     | 0.70               | 0.56               | .99672                     | 2.20               | 1.75               | .99459                     | 3.70               | 2.96               |
| .99884                     | 0.75               | 0.60               | .99665                     | 2.25               | 1.79               | .99452                     | 3.75               | 3.00               |
| .99877                     | 0.80               | 0.64               | .99658                     | 2.30               | 1.83               | .99445                     | 3.80               | 3.04               |
| .99869                     | 0.85               | 0.67               | .99651                     | 2.35               | 1.87               | .99438                     | 3.85               | 3.08               |
| .99861                     | 0.90               | 0.71               | .99643                     | 2.40               | 1.91               | .99431                     | 3.90               | 3.12               |
| .99854                     | 0.95               | 0.75               | .99636                     | 2.45               | 1.95               | .99424                     | 3.95               | 3.16               |
| .99849                     | 1.00               | 0.79               | .99629                     | 2.50               | 1.99               | .99417                     | 4.00               | 3.20               |
| .99842                     | 1.05               | 0.83               | .99622                     | 2.55               | 2.03               | .99410                     | 4.05               | 3.24               |
| .99834                     | 1.10               | 0.87               | .99615                     | 2.60               | 2.07               | .99403                     | 4.10               | 3.28               |
| .99827                     | 1.15               | 0.91               | .99607                     | 2.65               | 2.11               | .99397                     | 4.15               | 3.32               |
| .99819                     | 1.20               | 0.95               | .99600                     | 2.70               | 2.15               | .99390                     | 4.20               | 3.36               |
| .99812                     | 1.25               | 0.99               | .99593                     | 2.75               | 2.19               | .99383                     | 4.25               | 3.40               |
| .99805                     | 1.30               | 1.03               | .99586                     | 2.80               | 2.23               | .99376                     | 4.30               | 3.44               |
| .99797                     | 1.35               | 1.07               | .99579                     | 2.85               | 2.27               | .99369                     | 4.35               | 3.48               |
| .99790                     | 1.40               | 1.11               | .99571                     | 2.90               | 2.31               | .99363                     | 4.40               | 3.52               |
| .99782                     | 1.45               | 1.15               | .99564                     | 2.95               | 2.35               | .99356                     | 4.45               | 3.56               |

<sup>1</sup> From *Bulletin No. 107*, United States Department of Agriculture.



## PERCENTAGE OF ALCOHOL (CONTINUED)

| SPECIFIC GRAVITY AT 88° F. | ALCOHOL            |                    | SPECIFIC GRAVITY AT 88° F. | ALCOHOL            |                    | SPECIFIC GRAVITY AT 88° F. | ALCOHOL            |                    |
|----------------------------|--------------------|--------------------|----------------------------|--------------------|--------------------|----------------------------|--------------------|--------------------|
|                            | Per cent by volume | Per cent by weight |                            | Per cent by volume | Per cent by weight |                            | Per cent by volume | Per cent by weight |
| 0.99349                    | 4.50               | 3.60               | 0.99117                    | 6.25               | 5.00               | 0.98897                    | 8.00               | 6.42               |
| .99342                     | 4.55               | 3.64               | .99111                     | 6.30               | 5.05               | .98891                     | 8.05               | 6.46               |
| .99335                     | 4.60               | 3.68               | .99104                     | 6.35               | 5.09               | .98885                     | 8.10               | 6.50               |
| .99329                     | 4.65               | 3.72               | .99098                     | 6.40               | 5.13               | .98879                     | 8.15               | 6.54               |
| .99322                     | 4.70               | 3.76               | .99091                     | 6.45               | 5.17               | .98873                     | 8.20               | 6.58               |
| .99315                     | 4.75               | 3.80               | .99085                     | 6.50               | 5.21               | .98867                     | 8.25               | 6.62               |
| .99308                     | 4.80               | 3.84               | .99079                     | 6.55               | 5.25               | .98861                     | 8.30               | 6.67               |
| .99301                     | 4.85               | 3.88               | .99072                     | 6.60               | 5.29               | .98855                     | 8.35               | 6.71               |
| .99295                     | 4.90               | 3.92               | .99066                     | 6.65               | 5.33               | .98849                     | 8.40               | 6.75               |
| .99288                     | 4.95               | 3.96               | .99059                     | 6.70               | 5.37               | .98843                     | 8.45               | 6.79               |
| .99281                     | 5.00               | 4.00               | .99053                     | 6.75               | 5.41               | .98837                     | 8.50               | 6.83               |
| .99274                     | 5.05               | 4.04               | .99047                     | 6.80               | 5.45               | .98831                     | 8.55               | 6.87               |
| .99268                     | 5.10               | 4.08               | .99040                     | 6.85               | 5.49               | .98825                     | 8.60               | 6.91               |
| .99261                     | 5.15               | 4.12               | .99034                     | 6.90               | 5.53               | .98819                     | 8.65               | 6.95               |
| .99255                     | 5.20               | 4.16               | .99027                     | 6.95               | 5.57               | .98813                     | 8.70               | 6.99               |
| .99248                     | 5.25               | 4.20               | .99021                     | 7.00               | 5.61               | .98807                     | 8.75               | 7.03               |
| .99241                     | 5.30               | 4.24               | .99015                     | 7.05               | 5.65               | .98801                     | 8.80               | 7.07               |
| .99235                     | 5.35               | 4.28               | .99009                     | 7.10               | 5.69               | .98795                     | 8.85               | 7.11               |
| .99228                     | 5.40               | 4.32               | .99002                     | 7.15               | 5.73               | .98789                     | 8.90               | 7.15               |
| .99222                     | 5.45               | 4.36               | .98996                     | 7.20               | 5.77               | .98783                     | 8.95               | 7.19               |
| .99215                     | 5.50               | 4.40               | .98990                     | 7.25               | 5.81               | .98777                     | 9.00               | 7.23               |
| .99208                     | 5.55               | 4.44               | .98984                     | 7.30               | 5.86               | .98771                     | 9.05               | 7.27               |
| .99202                     | 5.60               | 4.48               | .98978                     | 7.35               | 5.90               | .98765                     | 9.10               | 7.31               |
| .99195                     | 5.65               | 4.52               | .98971                     | 7.40               | 5.94               | .98759                     | 9.15               | 7.35               |
| .99189                     | 5.70               | 4.56               | .98965                     | 7.45               | 5.98               | .98754                     | 9.20               | 7.39               |
| .99182                     | 5.75               | 4.60               | .98959                     | 7.50               | 6.02               | .98748                     | 9.25               | 7.43               |
| .99175                     | 5.80               | 4.64               | .98953                     | 7.55               | 6.06               | .98742                     | 9.30               | 7.48               |
| .99169                     | 5.85               | 4.68               | .98947                     | 7.60               | 6.10               | .98736                     | 9.35               | 7.52               |
| .99162                     | 5.90               | 4.72               | .98940                     | 7.65               | 6.14               | .98730                     | 9.40               | 7.56               |
| .99156                     | 5.95               | 4.76               | .98934                     | 7.70               | 6.18               | .98724                     | 9.45               | 7.60               |
| .99149                     | 6.00               | 4.80               | .98928                     | 7.75               | 6.22               | .98719                     | 9.50               | 7.64               |
| .99143                     | 6.05               | 4.84               | .98922                     | 7.80               | 6.26               | .98713                     | 9.55               | 7.68               |
| .99136                     | 6.10               | 4.88               | .98916                     | 7.85               | 6.30               | .98707                     | 9.60               | 7.72               |
| .99130                     | 6.15               | 4.92               | .98909                     | 7.90               | 6.34               | .98701                     | 9.65               | 7.76               |
| .99123                     | 6.20               | 4.96               | .98903                     | 7.95               | 6.38               | .98695                     | 9.70               | 7.80               |

## PERCENTAGE OF ALCOHOL (CONTINUED)

| SPECIFIC GRAVITY AT 88° F. | ALCOHOL            |                    | SPECIFIC GRAVITY AT 88° F. | ALCOHOL            |                    | SPECIFIC GRAVITY AT 88° F. | ALCOHOL            |                    |
|----------------------------|--------------------|--------------------|----------------------------|--------------------|--------------------|----------------------------|--------------------|--------------------|
|                            | Per cent by volume | Per cent by weight |                            | Per cent by volume | Per cent by weight |                            | Per cent by volume | Per cent by weight |
| 0.98689                    | 9.75               | 7.84               | 0.98491                    | 11.50              | 9.27               | 0.98299                    | 13.25              | 10.69              |
| .98683                     | 9.80               | 7.88               | .98485                     | 11.55              | 9.31               | .98294                     | 13.30              | 10.74              |
| .98678                     | 9.85               | 7.92               | .98479                     | 11.60              | 9.35               | .98289                     | 13.35              | 10.78              |
| .98672                     | 9.90               | 7.96               | .98474                     | 11.65              | 9.39               | .98283                     | 13.40              | 10.82              |
| .98666                     | 9.95               | 8.00               | .98468                     | 11.70              | 9.43               | .98278                     | 13.45              | 10.86              |
| .98660                     | 10.00              | 8.04               | .98463                     | 11.75              | 9.47               | .98273                     | 13.50              | 10.90              |
| .98654                     | 10.05              | 8.08               | .98457                     | 11.80              | 9.51               | .98267                     | 13.55              | 10.94              |
| .98649                     | 10.10              | 8.12               | .98452                     | 11.85              | 9.55               | .98262                     | 13.60              | 10.98              |
| .98643                     | 10.15              | 8.16               | .98446                     | 11.90              | 9.59               | .98256                     | 13.65              | 11.02              |
| .98637                     | 10.20              | 8.20               | .98441                     | 11.95              | 9.63               | .98251                     | 13.70              | 11.06              |
| .98632                     | 10.25              | 8.24               | .98435                     | 12.00              | 9.67               | .98246                     | 13.75              | 11.11              |
| .98626                     | 10.30              | 8.29               | .98430                     | 12.05              | 9.71               | .98240                     | 13.80              | 11.15              |
| .98620                     | 10.35              | 8.33               | .98424                     | 12.10              | 9.75               | .98235                     | 13.85              | 11.19              |
| .98614                     | 10.40              | 8.37               | .98419                     | 12.15              | 9.79               | .98230                     | 13.90              | 11.23              |
| .98609                     | 10.45              | 8.41               | .98413                     | 12.20              | 9.83               | .98224                     | 13.95              | 11.27              |
| .98603                     | 10.50              | 8.45               | .98408                     | 12.25              | 9.87               | .98219                     | 14.00              | 11.31              |
| .98597                     | 10.55              | 8.49               | .98402                     | 12.30              | 9.92               | .98214                     | 14.05              | 11.35              |
| .98592                     | 10.60              | 8.53               | .98397                     | 12.35              | 9.96               | .98209                     | 14.10              | 11.39              |
| .98586                     | 10.65              | 8.57               | .98391                     | 12.40              | 10.00              | .98203                     | 14.15              | 11.43              |
| .98580                     | 10.70              | 8.61               | .98386                     | 12.45              | 10.04              | .98198                     | 14.20              | 11.47              |
| .98575                     | 10.75              | 8.65               | .98381                     | 12.50              | 10.08              | .98193                     | 14.25              | 11.52              |
| .98569                     | 10.80              | 8.70               | .98375                     | 12.55              | 10.12              | .98188                     | 14.30              | 11.56              |
| .98563                     | 10.85              | 8.74               | .98370                     | 12.60              | 10.16              | .98182                     | 14.35              | 11.60              |
| .98557                     | 10.90              | 8.78               | .98364                     | 12.65              | 10.20              | .98177                     | 14.40              | 11.64              |
| .98552                     | 10.95              | 8.82               | .98359                     | 12.70              | 10.24              | .98172                     | 14.45              | 11.68              |
| .98546                     | 11.00              | 8.86               | .98353                     | 12.75              | 10.28              | .98167                     | 14.50              | 11.72              |
| .98540                     | 11.05              | 8.90               | .98348                     | 12.80              | 10.33              | .98161                     | 14.55              | 11.76              |
| .98535                     | 11.10              | 8.94               | .98342                     | 12.85              | 10.37              | .98156                     | 14.60              | 11.80              |
| .98529                     | 11.15              | 8.98               | .98337                     | 12.90              | 10.41              | .98151                     | 14.65              | 11.84              |
| .98524                     | 11.20              | 9.02               | .98331                     | 12.95              | 10.45              | .98146                     | 14.70              | 11.88              |
| .98518                     | 11.25              | 9.07               | .98326                     | 13.00              | 10.49              | .98140                     | 14.75              | 11.93              |
| .98513                     | 11.30              | 9.11               | .98321                     | 13.05              | 10.53              | .98135                     | 14.80              | 11.97              |
| .98507                     | 11.35              | 9.15               | .98315                     | 13.10              | 10.57              | .98130                     | 14.85              | 12.01              |
| .98502                     | 11.40              | 9.19               | .98310                     | 13.15              | 10.61              | .98125                     | 14.90              | 12.05              |
| .98496                     | 11.45              | 9.23               | .98305                     | 13.20              | 10.65              | .98119                     | 14.95              | 12.09              |

## PERCENTAGE OF ALCOHOL (CONTINUED)

| SPECIFIC GRAVITY AT 60° F. | ALCOHOL            |                    | SPECIFIC GRAVITY AT 60° F. | ALCOHOL            |                    | SPECIFIC GRAVITY AT 60° F. | ALCOHOL            |                    |
|----------------------------|--------------------|--------------------|----------------------------|--------------------|--------------------|----------------------------|--------------------|--------------------|
|                            | Per cent by volume | Per cent by weight |                            | Per cent by volume | Per cent by weight |                            | Per cent by volume | Per cent by weight |
| .098114                    | 15.00              | 12.13              | .097935                    | 16.75              | 13.57              | .097758                    | 18.50              | 15.02              |
| .098108                    | 15.05              | 12.17              | .097929                    | 16.80              | 13.62              | .097753                    | 18.55              | 15.06              |
| .098104                    | 15.10              | 12.21              | .097924                    | 16.85              | 13.66              | .097748                    | 18.60              | 15.10              |
| .098099                    | 15.15              | 12.25              | .097919                    | 16.90              | 13.70              | .097743                    | 18.65              | 15.14              |
| .098093                    | 15.20              | 12.29              | .097914                    | 16.95              | 13.74              | .097738                    | 18.70              | 15.18              |
| .098088                    | 15.25              | 12.33              | .097909                    | 17.00              | 13.78              | .097733                    | 18.75              | 15.22              |
| .098083                    | 15.30              | 12.38              | .097904                    | 17.05              | 13.82              | .097728                    | 18.80              | 15.27              |
| .098078                    | 15.35              | 12.42              | .097899                    | 17.10              | 13.86              | .097723                    | 18.85              | 15.31              |
| .098073                    | 15.40              | 12.46              | .097894                    | 17.15              | 13.90              | .097718                    | 18.90              | 15.38              |
| .098068                    | 15.45              | 12.50              | .097889                    | 17.20              | 13.94              | .097713                    | 18.95              | 15.39              |
| .098063                    | 15.50              | 12.54              | .097884                    | 17.25              | 13.98              | .097708                    | 19.00              | 15.43              |
| .098057                    | 15.55              | 12.58              | .097879                    | 17.30              | 14.03              | .097703                    | 19.05              | 15.47              |
| .098052                    | 15.60              | 12.62              | .097874                    | 17.35              | 14.07              | .097698                    | 19.10              | 15.51              |
| .098047                    | 15.65              | 12.66              | .097869                    | 17.40              | 14.11              | .097693                    | 19.15              | 15.55              |
| .098042                    | 15.70              | 12.70              | .097864                    | 17.45              | 14.15              | .097688                    | 19.20              | 15.59              |
| .098037                    | 15.75              | 12.75              | .097859                    | 17.50              | 14.19              | .097683                    | 19.25              | 15.63              |
| .098032                    | 15.80              | 12.79              | .097853                    | 17.55              | 14.23              | .097678                    | 19.30              | 15.68              |
| .098026                    | 15.85              | 12.83              | .097848                    | 17.60              | 14.27              | .097673                    | 19.35              | 15.72              |
| .098021                    | 15.90              | 12.87              | .097843                    | 17.65              | 14.31              | .097668                    | 19.40              | 15.76              |
| .098016                    | 15.95              | 12.91              | .097838                    | 17.70              | 14.35              | .097663                    | 19.45              | 15.80              |
| .098011                    | 16.00              | 12.95              | .097833                    | 17.75              | 14.40              | .097658                    | 19.50              | 15.84              |
| .098005                    | 16.05              | 12.99              | .097828                    | 17.80              | 14.44              | .097653                    | 19.55              | 15.88              |
| .098001                    | 16.10              | 13.03              | .097823                    | 17.85              | 14.48              | .097648                    | 19.60              | 15.93              |
| .097996                    | 16.15              | 13.08              | .097818                    | 17.90              | 14.52              | .097643                    | 19.65              | 15.97              |
| .097991                    | 16.20              | 13.12              | .097813                    | 17.95              | 14.56              | .097638                    | 19.70              | 16.01              |
| .097986                    | 16.25              | 13.16              | .097808                    | 18.00              | 14.60              | .097633                    | 19.75              | 16.05              |
| .097980                    | 16.30              | 13.20              | .097803                    | 18.05              | 14.64              | .097628                    | 19.80              | 16.09              |
| .097975                    | 16.35              | 13.24              | .097798                    | 18.10              | 14.68              | .097623                    | 19.85              | 16.14              |
| .097970                    | 16.40              | 13.29              | .097793                    | 18.15              | 14.73              | .097618                    | 19.90              | 16.18              |
| .097965                    | 16.45              | 13.33              | .097788                    | 18.20              | 14.77              | .097613                    | 19.95              | 16.22              |
| .097960                    | 16.50              | 13.37              | .097783                    | 18.25              | 14.81              | .097608                    | 20.00              | 16.26              |
| .097955                    | 16.55              | 13.41              | .097778                    | 18.30              | 14.85              | .097603                    | 20.05              | 16.30              |
| .097950                    | 16.60              | 13.45              | .097773                    | 18.35              | 14.89              | .097598                    | 20.10              | 16.34              |
| .097945                    | 16.65              | 13.49              | .097768                    | 18.40              | 14.94              | .097593                    | 20.15              | 16.38              |
| .097940                    | 16.70              | 13.53              | .097763                    | 18.45              | 14.98              | .097588                    | 20.20              | 16.42              |

## PERCENTAGE OF ALCOHOL (CONTINUED)

| SPECIFIC GRAVITY AT 88° F. | ALCOHOL            |                    | SPECIFIC GRAVITY AT 88° F. | ALCOHOL            |                    | SPECIFIC GRAVITY AT 88° F. | ALCOHOL            |                    |
|----------------------------|--------------------|--------------------|----------------------------|--------------------|--------------------|----------------------------|--------------------|--------------------|
|                            | Per cent by volume | Per cent by weight |                            | Per cent by volume | Per cent by weight |                            | Per cent by volume | Per cent by weight |
| .97583                     | 20.25              | 16.46              | .97406                     | 22.00              | 17.92              | .97227                     | 23.75              | 19.38              |
| .97578                     | 20.30              | 16.51              | .97401                     | 22.05              | 17.96              | .97222                     | 23.80              | 19.42              |
| .97573                     | 20.35              | 16.58              | .97396                     | 22.10              | 18.00              | .97216                     | 23.85              | 19.46              |
| .97568                     | 20.40              | 16.59              | .97391                     | 22.15              | 18.05              | .97211                     | 23.90              | 19.51              |
| .97563                     | 20.45              | 16.63              | .97386                     | 22.20              | 18.09              | .97206                     | 23.95              | 19.55              |
| .97558                     | 20.50              | 16.67              | .97381                     | 22.25              | 18.13              | .97201                     | 24.00              | 19.59              |
| .97552                     | 20.55              | 16.71              | .97375                     | 22.30              | 18.17              | .97196                     | 24.05              | 19.63              |
| .97547                     | 20.60              | 16.75              | .97370                     | 22.35              | 18.21              | .97191                     | 24.10              | 19.67              |
| .97542                     | 20.65              | 16.80              | .97365                     | 22.40              | 18.26              | .97185                     | 24.15              | 19.72              |
| .97537                     | 20.70              | 16.84              | .97360                     | 22.45              | 18.30              | .97180                     | 24.20              | 19.76              |
| .97532                     | 20.75              | 16.88              | .97355                     | 22.50              | 18.34              | .97175                     | 24.25              | 19.80              |
| .97527                     | 20.80              | 16.92              | .97350                     | 22.55              | 18.38              | .97170                     | 24.30              | 19.84              |
| .97522                     | 20.85              | 16.96              | .97345                     | 22.60              | 18.42              | .97165                     | 24.35              | 19.88              |
| .97517                     | 20.90              | 17.01              | .97340                     | 22.65              | 18.47              | .97159                     | 24.40              | 19.93              |
| .97512                     | 20.95              | 17.05              | .97335                     | 22.70              | 18.51              | .97154                     | 24.45              | 19.97              |
| .97507                     | 21.00              | 17.09              | .97330                     | 22.75              | 18.55              | .97149                     | 24.50              | 20.01              |
| .97502                     | 21.05              | 17.13              | .97324                     | 22.80              | 18.59              | .97144                     | 24.55              | 20.05              |
| .97497                     | 21.10              | 17.17              | .97319                     | 22.85              | 18.63              | .97139                     | 24.60              | 20.09              |
| .97492                     | 21.15              | 17.22              | .97314                     | 22.90              | 18.68              | .97133                     | 24.65              | 20.14              |
| .97487                     | 21.20              | 17.26              | .97309                     | 22.95              | 18.72              | .97128                     | 24.70              | 20.18              |
| .97482                     | 21.25              | 17.30              | .97304                     | 23.00              | 18.76              | .97123                     | 24.75              | 20.22              |
| .97477                     | 21.30              | 17.34              | .97299                     | 23.05              | 18.80              | .97118                     | 24.80              | 20.26              |
| .97472                     | 21.35              | 17.38              | .97294                     | 23.10              | 18.84              | .97113                     | 24.85              | 20.30              |
| .97467                     | 21.40              | 17.43              | .97289                     | 23.15              | 18.88              | .97107                     | 24.90              | 20.35              |
| .97462                     | 21.45              | 17.47              | .97283                     | 23.20              | 18.92              | .97102                     | 24.95              | 20.39              |
| .97457                     | 21.50              | 17.51              | .97278                     | 23.25              | 18.96              | .97097                     | 25.00              | 20.43              |
| .97451                     | 21.55              | 17.55              | .97273                     | 23.30              | 19.01              | .97092                     | 25.05              | 20.47              |
| .97446                     | 21.60              | 17.59              | .97268                     | 23.35              | 19.05              | .97086                     | 25.10              | 20.51              |
| .97441                     | 21.65              | 17.63              | .97263                     | 23.40              | 19.09              | .97081                     | 25.15              | 20.56              |
| .97436                     | 21.70              | 17.67              | .97258                     | 23.45              | 19.13              | .97076                     | 25.20              | 20.60              |
| .97431                     | 21.75              | 17.71              | .97253                     | 23.50              | 19.17              | .97071                     | 25.25              | 20.64              |
| .97426                     | 21.80              | 17.76              | .97247                     | 23.55              | 19.21              | .97065                     | 25.30              | 20.68              |
| .97421                     | 21.85              | 17.80              | .97242                     | 23.60              | 19.25              | .97060                     | 25.35              | 20.72              |
| .97416                     | 21.90              | 17.84              | .97237                     | 23.65              | 19.30              | .97055                     | 25.40              | 20.77              |
| .97411                     | 21.95              | 17.88              | .97232                     | 23.70              | 19.34              | .97049                     | 25.45              | 20.81              |

PERCENTAGE OF ALCOHOL

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PERCENTAGE OF ALCOHOL (CONTINUED)

| SPECIFIC GRAVITY AT 60° F. | ALCOHOL            |                    | SPECIFIC GRAVITY AT 60° F. | ALCOHOL            |                    | SPECIFIC GRAVITY AT 60° F. | ALCOHOL            |                    |
|----------------------------|--------------------|--------------------|----------------------------|--------------------|--------------------|----------------------------|--------------------|--------------------|
|                            | Per cent by volume | Per cent by weight |                            | Per cent by volume | Per cent by weight |                            | Per cent by volume | Per cent by weight |
| 0.97044                    | 25.50              | 20.85              | 0.96855                    | 27.25              | 22.33              | 0.96658                    | 29.00              | 23.81              |
| .97039                     | 25.55              | 20.89              | .96850                     | 27.30              | 22.37              | .96652                     | 29.05              | 23.85              |
| .97033                     | 25.60              | 20.93              | .96844                     | 27.35              | 22.41              | .96646                     | 29.10              | 23.89              |
| .97028                     | 25.65              | 20.98              | .96839                     | 27.40              | 22.45              | .96640                     | 29.15              | 23.94              |
| .97023                     | 25.70              | 21.02              | .96833                     | 27.45              | 22.50              | .96635                     | 29.20              | 23.98              |
| .97018                     | 25.75              | 21.06              | .96828                     | 27.50              | 22.54              | .96629                     | 29.25              | 24.02              |
| .97012                     | 25.80              | 21.10              | .96822                     | 27.55              | 22.58              | .96623                     | 29.30              | 24.06              |
| .97007                     | 25.85              | 21.14              | .96816                     | 27.60              | 22.62              | .96617                     | 29.35              | 24.10              |
| .97001                     | 25.90              | 21.19              | .96811                     | 27.65              | 22.67              | .96611                     | 29.40              | 24.15              |
| .96996                     | 25.95              | 21.23              | .96805                     | 27.70              | 22.71              | .96605                     | 29.45              | 24.19              |
| .96991                     | 26.00              | 21.27              | .96800                     | 27.75              | 22.75              | .96600                     | 29.50              | 24.23              |
| .96986                     | 26.05              | 21.31              | .96794                     | 27.80              | 22.79              | .96594                     | 29.55              | 24.27              |
| .96980                     | 26.10              | 21.35              | .96789                     | 27.85              | 22.83              | .96588                     | 29.60              | 24.32              |
| .96975                     | 26.15              | 21.40              | .96783                     | 27.90              | 22.88              | .96582                     | 29.65              | 24.36              |
| .96969                     | 26.20              | 21.44              | .96778                     | 27.95              | 22.92              | .96576                     | 29.70              | 24.40              |
| .96964                     | 26.25              | 21.48              | .96772                     | 28.00              | 22.96              | .96570                     | 29.75              | 24.45              |
| .96959                     | 26.30              | 21.52              | .96766                     | 28.05              | 23.00              | .96564                     | 29.80              | 24.49              |
| .96953                     | 26.35              | 21.56              | .96761                     | 28.10              | 23.04              | .96559                     | 29.85              | 24.53              |
| .96949                     | 26.40              | 21.61              | .96755                     | 28.15              | 23.09              | .96553                     | 29.90              | 24.57              |
| .96942                     | 26.45              | 21.65              | .96749                     | 28.20              | 23.13              | .96547                     | 29.95              | 24.62              |
| .96937                     | 26.50              | 21.69              | .96744                     | 28.25              | 23.17              | .96541                     | 30.00              | 24.66              |
| .96932                     | 26.55              | 21.73              | .96738                     | 28.30              | 23.21              | .96535                     | 30.05              | 24.70              |
| .96926                     | 26.60              | 21.77              | .96732                     | 28.35              | 23.25              | .96529                     | 30.10              | 24.74              |
| .96921                     | 26.65              | 21.82              | .96726                     | 28.40              | 23.30              | .96523                     | 30.15              | 24.79              |
| .96915                     | 26.70              | 21.86              | .96721                     | 28.45              | 23.34              | .96517                     | 30.20              | 24.83              |
| .96910                     | 26.75              | 21.90              | .96715                     | 28.50              | 23.38              | .96511                     | 30.25              | 24.87              |
| .96905                     | 26.80              | 21.94              | .96709                     | 28.55              | 23.42              | .96505                     | 30.30              | 24.91              |
| .96899                     | 26.85              | 21.98              | .96704                     | 28.60              | 23.47              | .96499                     | 30.35              | 24.95              |
| .96894                     | 26.90              | 22.03              | .96698                     | 28.65              | 23.51              | .96493                     | 30.40              | 25.00              |
| .96888                     | 26.95              | 22.07              | .96692                     | 28.70              | 23.55              | .96487                     | 30.45              | 25.04              |
| .96883                     | 27.00              | 22.11              | .96687                     | 28.75              | 23.60              | .96481                     | 30.50              | 25.08              |
| .96877                     | 27.05              | 22.15              | .96681                     | 28.80              | 23.64              | .96475                     | 30.55              | 25.12              |
| .96872                     | 27.10              | 22.20              | .96675                     | 28.85              | 23.68              | .96469                     | 30.60              | 25.17              |
| .96866                     | 27.15              | 22.24              | .96669                     | 28.90              | 23.72              | .96463                     | 30.65              | 25.21              |
| .96861                     | 27.20              | 22.28              | .96664                     | 28.95              | 23.77              | .96457                     | 30.70              | 25.25              |

## PERCENTAGE OF ALCOHOL (CONTINUED)

| SPECIFIC GRAVITY AT 60° F. | ALCOHOL            |                    | SPECIFIC GRAVITY AT 60° F. | ALCOHOL            |                    | SPECIFIC GRAVITY AT 60° F. | ALCOHOL            |                    |
|----------------------------|--------------------|--------------------|----------------------------|--------------------|--------------------|----------------------------|--------------------|--------------------|
|                            | Per cent by volume | Per cent by weight |                            | Per cent by volume | Per cent by weight |                            | Per cent by volume | Per cent by weight |
| 0.96451                    | 30.75              | 25.30              | 0.96235                    | 32.50              | 26.80              | 0.96010                    | 34.25              | 28.31              |
| .96445                     | 30.80              | 25.34              | .96229                     | 32.55              | 26.84              | .96003                     | 34.30              | 28.35              |
| .96439                     | 30.85              | 25.38              | .96222                     | 32.60              | 26.89              | .95996                     | 34.35              | 28.39              |
| .96433                     | 30.90              | 25.42              | .96216                     | 32.65              | 26.93              | .95990                     | 34.40              | 28.43              |
| .96427                     | 30.95              | 25.47              | .96210                     | 32.70              | 26.97              | .95983                     | 34.45              | 28.48              |
| .96421                     | 31.00              | 25.51              | .96204                     | 32.75              | 27.02              | .95977                     | 34.50              | 28.52              |
| .96415                     | 31.05              | 25.55              | .96197                     | 32.80              | 27.06              | .95970                     | 34.55              | 28.56              |
| .96409                     | 31.10              | 25.60              | .96191                     | 32.85              | 27.10              | .95963                     | 34.60              | 28.61              |
| .96403                     | 31.15              | 25.64              | .96185                     | 32.90              | 27.14              | .95957                     | 34.65              | 28.65              |
| .96396                     | 31.20              | 25.68              | .96178                     | 32.95              | 27.19              | .95950                     | 34.70              | 28.70              |
| .96390                     | 31.25              | 25.73              | .96172                     | 33.00              | 27.23              | .95943                     | 34.75              | 28.74              |
| .96384                     | 31.30              | 25.77              | .96166                     | 33.05              | 27.27              | .95937                     | 34.80              | 28.78              |
| .96378                     | 31.35              | 25.81              | .96159                     | 33.10              | 27.32              | .95930                     | 34.85              | 28.83              |
| .96372                     | 31.40              | 25.85              | .96153                     | 33.15              | 27.36              | .95923                     | 34.90              | 28.87              |
| .96366                     | 31.45              | 25.90              | .96146                     | 33.20              | 27.40              | .95917                     | 34.95              | 28.92              |
| .96360                     | 31.50              | 25.94              | .96140                     | 33.25              | 27.45              | .95910                     | 35.00              | 28.96              |
| .96353                     | 31.55              | 25.98              | .96133                     | 33.30              | 27.49              | .95903                     | 35.05              | 29.00              |
| .96347                     | 31.60              | 26.03              | .96127                     | 33.35              | 27.53              | .95896                     | 35.10              | 29.05              |
| .96341                     | 31.65              | 26.07              | .96120                     | 33.40              | 27.57              | .95889                     | 35.15              | 29.09              |
| .96335                     | 31.70              | 26.11              | .96114                     | 33.45              | 27.62              | .95883                     | 35.20              | 29.13              |
| .96329                     | 31.75              | 26.16              | .96108                     | 33.50              | 27.66              | .95876                     | 35.25              | 29.18              |
| .96323                     | 31.80              | 26.20              | .96101                     | 33.55              | 27.70              | .95869                     | 35.30              | 29.22              |
| .96316                     | 31.85              | 26.24              | .96095                     | 33.60              | 27.75              | .95862                     | 35.35              | 29.26              |
| .96310                     | 31.90              | 26.28              | .96088                     | 33.65              | 27.79              | .95855                     | 35.40              | 29.30              |
| .96304                     | 31.95              | 26.33              | .96082                     | 33.70              | 27.83              | .95848                     | 35.45              | 29.35              |
| .96298                     | 32.00              | 26.37              | .96075                     | 33.75              | 27.88              | .95842                     | 35.50              | 29.39              |
| .96292                     | 32.05              | 26.41              | .96069                     | 33.80              | 27.92              | .95835                     | 35.55              | 29.43              |
| .96285                     | 32.10              | 26.46              | .96062                     | 33.85              | 27.96              | .95828                     | 35.60              | 29.48              |
| .96279                     | 32.15              | 26.50              | .96056                     | 33.90              | 28.00              | .95821                     | 35.65              | 29.52              |
| .96273                     | 32.20              | 26.54              | .96049                     | 33.95              | 28.05              | .95814                     | 35.70              | 29.57              |
| .96267                     | 32.25              | 26.59              | .96043                     | 34.00              | 28.09              | .95807                     | 35.75              | 29.61              |
| .96260                     | 32.30              | 26.63              | .96036                     | 34.05              | 28.13              | .95800                     | 35.80              | 29.65              |
| .96254                     | 32.35              | 26.67              | .96030                     | 34.10              | 28.18              | .95794                     | 35.85              | 29.70              |
| .96248                     | 32.40              | 26.71              | .96023                     | 34.15              | 28.22              | .95787                     | 35.90              | 29.74              |
| .96241                     | 32.45              | 26.76              | .96016                     | 34.20              | 28.26              | .95780                     | 35.95              | 29.79              |

PERCENTAGE OF ALCOHOL (CONTINUED)

| SPECIFIC GRAVITY AT 88° F. | ALCOHOL            |                    | SPECIFIC GRAVITY AT 88° F. | ALCOHOL            |                    | SPECIFIC GRAVITY AT 88° F. | ALCOHOL            |                    |
|----------------------------|--------------------|--------------------|----------------------------|--------------------|--------------------|----------------------------|--------------------|--------------------|
|                            | Per cent by volume | Per cent by weight |                            | Per cent by volume | Per cent by weight |                            | Per cent by volume | Per cent by weight |
| 0.95773                    | 36.00              | 29.83              | 0.95523                    | 37.75              | 31.36              | 0.95262                    | 39.50              | 32.90              |
| .95766                     | 36.05              | 29.87              | .95516                     | 37.80              | 31.40              | .95254                     | 39.55              | 32.95              |
| .95759                     | 36.10              | 29.92              | .95509                     | 37.85              | 31.45              | .95246                     | 39.60              | 32.99              |
| .95752                     | 36.15              | 29.96              | .95502                     | 37.90              | 31.49              | .95239                     | 39.65              | 33.04              |
| .95745                     | 36.20              | 30.00              | .95494                     | 37.95              | 31.54              | .95231                     | 39.70              | 33.08              |
| .95738                     | 36.25              | 30.05              | .95487                     | 38.00              | 31.58              | .95223                     | 39.75              | 33.13              |
| .95731                     | 36.30              | 30.09              | .95480                     | 38.05              | 31.63              | .95216                     | 39.80              | 33.17              |
| .95724                     | 36.35              | 30.13              | .95472                     | 38.10              | 31.67              | .95208                     | 39.85              | 33.22              |
| .95717                     | 36.40              | 30.17              | .95465                     | 38.15              | 31.72              | .95200                     | 39.90              | 33.27              |
| .95710                     | 36.45              | 30.22              | .95457                     | 38.20              | 31.76              | .95193                     | 39.95              | 33.31              |
| .95703                     | 36.50              | 30.26              | .95450                     | 38.25              | 31.81              | .95185                     | 40.00              | 33.35              |
| .95695                     | 36.55              | 30.30              | .95442                     | 38.30              | 31.85              | .95177                     | 40.05              | 33.39              |
| .95688                     | 36.60              | 30.35              | .95435                     | 38.35              | 31.90              | .95169                     | 40.10              | 33.44              |
| .95681                     | 36.65              | 30.39              | .95427                     | 38.40              | 31.94              | .95161                     | 40.15              | 33.48              |
| .95674                     | 36.70              | 30.44              | .95420                     | 38.45              | 31.99              | .95154                     | 40.20              | 33.53              |
| .95667                     | 36.75              | 30.48              | .95413                     | 38.50              | 32.03              | .95146                     | 40.25              | 33.57              |
| .95660                     | 36.80              | 30.52              | .95405                     | 38.55              | 32.07              | .95138                     | 40.30              | 33.61              |
| .95653                     | 36.85              | 30.57              | .95398                     | 38.60              | 32.12              | .95130                     | 40.35              | 33.66              |
| .95646                     | 36.90              | 30.61              | .95390                     | 38.65              | 32.16              | .95122                     | 40.40              | 33.70              |
| .95639                     | 36.95              | 30.66              | .95383                     | 38.70              | 32.20              | .95114                     | 40.45              | 33.75              |
| .95632                     | 37.00              | 30.70              | .95375                     | 38.75              | 32.25              | .95107                     | 40.50              | 33.79              |
| .95625                     | 37.05              | 30.74              | .95368                     | 38.80              | 32.29              | .95099                     | 40.55              | 33.84              |
| .95618                     | 37.10              | 30.79              | .95360                     | 38.85              | 32.33              | .95091                     | 40.60              | 33.88              |
| .95610                     | 37.15              | 30.83              | .95353                     | 38.90              | 32.37              | .95083                     | 40.65              | 33.93              |
| .95603                     | 37.20              | 30.88              | .95345                     | 38.95              | 32.42              | .95075                     | 40.70              | 33.97              |
| .95596                     | 37.25              | 30.92              | .95338                     | 39.00              | 32.46              | .95067                     | 40.75              | 34.02              |
| .95589                     | 37.30              | 30.96              | .95330                     | 39.05              | 32.50              | .95059                     | 40.80              | 34.06              |
| .95581                     | 37.35              | 31.01              | .95323                     | 39.10              | 32.55              | .95052                     | 40.85              | 34.11              |
| .95574                     | 37.40              | 31.05              | .95315                     | 39.15              | 32.59              | .95044                     | 40.90              | 34.15              |
| .95567                     | 37.45              | 31.10              | .95307                     | 39.20              | 32.64              | .95036                     | 40.95              | 34.20              |
| .95560                     | 37.50              | 31.14              | .95300                     | 39.25              | 32.68              | .95028                     | 41.00              | 34.24              |
| .95552                     | 37.55              | 31.18              | .95292                     | 39.30              | 32.72              | .95020                     | 41.05              | 34.28              |
| .95545                     | 37.60              | 31.23              | .95284                     | 39.35              | 32.77              | .95012                     | 41.10              | 34.33              |
| .95538                     | 37.65              | 31.27              | .95277                     | 39.40              | 32.81              | .95004                     | 41.15              | 34.37              |
| .95531                     | 37.70              | 31.32              | .95269                     | 39.45              | 32.86              | .94996                     | 41.20              | 34.42              |

## PERCENTAGE OF ALCOHOL (CONTINUED)

| SPECIFIC GRAVITY AT 60° F. | ALCOHOL            |                    | SPECIFIC GRAVITY AT 60° F. | ALCOHOL            |                    | SPECIFIC GRAVITY AT 60° F. | ALCOHOL            |                    |
|----------------------------|--------------------|--------------------|----------------------------|--------------------|--------------------|----------------------------|--------------------|--------------------|
|                            | Per cent by volume | Per cent by weight |                            | Per cent by volume | Per cent by weight |                            | Per cent by volume | Per cent by weight |
| 0.94988                    | 41.25              | 34.46              | 0.94704                    | 43.00              | 36.03              | 0.94407                    | 44.75              | 37.62              |
| .94980                     | 41.30              | 34.50              | .94696                     | 43.05              | 36.08              | .94398                     | 44.80              | 37.66              |
| .94972                     | 41.35              | 34.55              | .94687                     | 43.10              | 36.12              | .94390                     | 44.85              | 37.71              |
| .94964                     | 41.40              | 34.59              | .94679                     | 43.15              | 36.17              | .94381                     | 44.90              | 37.76              |
| .94956                     | 41.45              | 34.64              | .94670                     | 43.20              | 36.21              | .94373                     | 44.95              | 37.80              |
| .94948                     | 41.50              | 34.68              | .94662                     | 43.25              | 36.23              | .94364                     | 45.00              | 37.84              |
| .94940                     | 41.55              | 34.73              | .94654                     | 43.30              | 36.30              | .94355                     | 45.05              | 37.89              |
| .94932                     | 41.60              | 34.77              | .94645                     | 43.35              | 36.35              | .94346                     | 45.10              | 37.93              |
| .94924                     | 41.65              | 34.82              | .94637                     | 43.40              | 36.39              | .94338                     | 45.15              | 37.98              |
| .94916                     | 41.70              | 34.86              | .94628                     | 43.45              | 36.44              | .94329                     | 45.20              | 38.02              |
| .94908                     | 41.75              | 34.91              | .94620                     | 43.50              | 36.48              | .94320                     | 45.25              | 38.07              |
| .94900                     | 41.80              | 34.95              | .94612                     | 43.55              | 36.53              | .94311                     | 45.30              | 38.12              |
| .94892                     | 41.85              | 35.00              | .94603                     | 43.60              | 36.57              | .94302                     | 45.35              | 38.16              |
| .94884                     | 41.90              | 35.04              | .94595                     | 43.65              | 36.62              | .94294                     | 45.40              | 38.21              |
| .94876                     | 41.95              | 35.09              | .94586                     | 43.70              | 36.66              | .94285                     | 45.45              | 38.25              |
| .94868                     | 42.00              | 35.13              | .94578                     | 43.75              | 36.71              | .94276                     | 45.50              | 38.30              |
| .94860                     | 42.05              | 35.18              | .94570                     | 43.80              | 36.75              | .94267                     | 45.55              | 38.35              |
| .94852                     | 42.10              | 35.22              | .94561                     | 43.85              | 36.80              | .94258                     | 45.60              | 38.39              |
| .94843                     | 42.15              | 35.27              | .94553                     | 43.90              | 36.84              | .94250                     | 45.65              | 38.44              |
| .94835                     | 42.20              | 35.31              | .94544                     | 43.95              | 36.89              | .94241                     | 45.70              | 38.48              |
| .94827                     | 42.25              | 35.36              | .94536                     | 44.00              | 36.93              | .94232                     | 45.75              | 38.53              |
| .94810                     | 42.30              | 35.40              | .94527                     | 44.05              | 36.98              | .94223                     | 45.80              | 38.57              |
| .94811                     | 42.35              | 35.45              | .94519                     | 44.10              | 37.02              | .94214                     | 45.85              | 38.62              |
| .94802                     | 42.40              | 35.49              | .94510                     | 44.15              | 37.07              | .94206                     | 45.90              | 38.66              |
| .94794                     | 42.45              | 35.54              | .94502                     | 44.20              | 37.11              | .94197                     | 45.95              | 38.71              |
| .94786                     | 42.50              | 35.58              | .94493                     | 44.25              | 37.16              | .94188                     | 46.00              | 38.75              |
| .94778                     | 42.55              | 35.63              | .94484                     | 44.30              | 37.21              | .94179                     | 46.05              | 38.80              |
| .94770                     | 42.60              | 35.67              | .94476                     | 44.35              | 37.25              | .94170                     | 46.10              | 38.84              |
| .94761                     | 42.65              | 35.72              | .94467                     | 44.40              | 37.30              | .94161                     | 46.15              | 38.89              |
| .94753                     | 42.70              | 35.76              | .94459                     | 44.45              | 37.34              | .94152                     | 46.20              | 38.93              |
| .94745                     | 42.75              | 35.81              | .94450                     | 44.50              | 37.39              | .94143                     | 46.25              | 38.98              |
| .94737                     | 42.80              | 35.85              | .94441                     | 44.55              | 37.44              | .94134                     | 46.30              | 39.03              |
| .94729                     | 42.85              | 35.90              | .94433                     | 44.60              | 37.48              | .94125                     | 46.35              | 39.07              |
| .94720                     | 42.90              | 35.94              | .94424                     | 44.65              | 37.53              | .94116                     | 46.40              | 39.12              |
| .94712                     | 42.95              | 35.99              | .94416                     | 44.70              | 37.57              | .94107                     | 46.45              | 39.16              |



PERCENTAGE OF ALCOHOL (CONCLUDED)

| SPECIFIC GRAVITY AT 60° F. | ALCOHOL            |                    | SPECIFIC GRAVITY AT 60° F. | ALCOHOL            |                    | SPECIFIC GRAVITY AT 60° F. | ALCOHOL            |                    |
|----------------------------|--------------------|--------------------|----------------------------|--------------------|--------------------|----------------------------|--------------------|--------------------|
|                            | Per cent by volume | Per cent by weight |                            | Per cent by volume | Per cent by weight |                            | Per cent by volume | Per cent by weight |
| 0.94098                    | 46.50              | 39.21              | 0.93870                    | 47.75              | 40.37              | 0.93636                    | 49.00              | 41.52              |
| .94089                     | 46.55              | 39.26              | .93861                     | 47.80              | 40.41              | .93626                     | 49.05              | 41.57              |
| .94080                     | 46.60              | 39.30              | .93852                     | 47.85              | 40.46              | .93617                     | 49.10              | 41.61              |
| .94071                     | 46.65              | 39.35              | .93842                     | 47.90              | 40.51              | .93607                     | 49.15              | 41.66              |
| .94062                     | 46.70              | 39.39              | .93833                     | 47.95              | 40.55              | .93598                     | 49.20              | 41.71              |
| .94053                     | 46.75              | 39.44              | .93824                     | 48.00              | 40.60              | .93588                     | 49.25              | 41.76              |
| .94044                     | 46.80              | 39.49              | .93815                     | 48.05              | 40.65              | .93578                     | 49.30              | 41.80              |
| .94035                     | 46.85              | 39.53              | .93805                     | 48.10              | 40.69              | .93569                     | 49.35              | 41.85              |
| .94026                     | 46.90              | 39.58              | .93796                     | 48.15              | 40.74              | .93559                     | 49.40              | 41.90              |
| .94017                     | 46.95              | 39.62              | .93786                     | 48.20              | 40.78              | .93550                     | 49.45              | 41.94              |
| .94008                     | 47.00              | 39.67              | .93777                     | 48.25              | 40.83              | .93540                     | 49.50              | 41.99              |
| .93999                     | 47.05              | 39.72              | .93768                     | 48.30              | 40.88              | .93530                     | 49.55              | 42.04              |
| .93990                     | 47.10              | 39.76              | .93758                     | 48.35              | 40.92              | .93521                     | 49.60              | 42.08              |
| .93980                     | 47.15              | 39.81              | .93749                     | 48.40              | 40.97              | .93511                     | 49.65              | 42.13              |
| .93971                     | 47.20              | 39.85              | .93739                     | 48.45              | 41.01              | .93502                     | 49.70              | 42.18              |
| .93962                     | 47.25              | 39.90              | .93730                     | 48.50              | 41.06              | .93492                     | 49.75              | 42.23              |
| .93953                     | 47.30              | 39.95              | .93721                     | 48.55              | 41.11              | .93482                     | 49.80              | 42.27              |
| .93944                     | 47.35              | 39.99              | .93711                     | 48.60              | 41.15              | .93473                     | 49.85              | 42.32              |
| .93934                     | 47.40              | 40.04              | .93702                     | 48.65              | 41.20              | .93463                     | 49.90              | 42.37              |
| .93925                     | 47.45              | 40.08              | .93692                     | 48.70              | 41.24              | .93454                     | 49.95              | 42.41              |
| .93916                     | 47.50              | 40.13              | .93683                     | 48.75              | 41.29              |                            |                    |                    |
| .93906                     | 47.55              | 40.18              | .93679                     | 48.80              | 41.34              |                            |                    |                    |
| .93898                     | 47.60              | 40.22              | .93664                     | 48.85              | 41.38              |                            |                    |                    |
| .93888                     | 47.65              | 40.27              | .93655                     | 48.90              | 41.43              |                            |                    |                    |
| .93879                     | 47.70              | 40.32              | .93645                     | 48.95              | 41.47              |                            |                    |                    |

## SECTION XII

### DETECTION OF COAL-TAR DYE

The use of coal-tar dyes in food and drink, while very general, is quite unnecessary, and frequently constitutes fraudulent adulteration. These dyes can usually be detected by the following methods:

**Double-Dyeing Process.** *Sostegni and Carpentieri.* If a solid or semisolid, dissolve 10 to 20 g. of the sample in 100 cc. of water. If a liquid, use from 50 to 100 cc., depending upon the intensity of the color. Acidify with 2 to 4 cc. of 10 per cent HCl. Boil nun's veiling or other white woolen cloth in very dilute KOH or strong soapsuds and wash thoroughly. Boil a piece about 10 cm. square in the dissolved sample until it has been well colored. This usually takes from five to ten minutes.

Remove the cloth, wash in cold water, and boil in 2 per cent HCl. After thorough rinsing dissolve the color in hot dilute  $\text{NH}_4\text{OH}$  (1 to 50). Remove the cloth and throw it away.

Add a slight excess of HCl to the ammonia solution. Immerse in this a second and smaller piece of cloth ( $2 \times 3$  cm.) and boil.

If the dye is of vegetable origin, the second piece of wool will be practically uncolored in the ammonia solution; if it is of coal-tar origin, it will take a decided tone, — red, pink, yellow, green, etc., — depending upon the color of the sample tested.

**Arata's Method.** Dissolve 20 to 30 g. of the sample in 100 cc. of water and 10 cc. of a 10 per cent solution of  $\text{HKSO}_4$ . In this mixture boil a small piece of cloth which has been previously boiled in dilute  $\text{NaOH}$  and thoroughly washed in water. Remove, wash in water, and dry between filter or blotting papers.

If the coloring matter is entirely of vegetable origin, the wool will be uncolored or will take a faint pink or brown, which is changed to green or yellow by ammonia and not restored by washing.

In addition, double dye, as indicated in the previous method. If the wool is still uncolored, the dye is of vegetable origin.

Nun's veiling is a very suitable cloth for these experiments. In removing the natural wool fat many students make the mistake of boiling it in too strong caustic. A 1 per cent solution is sufficiently strong.

Cochineal and some vegetable colors dye wool directly, hence the necessity of double dyeing.

Common substances which furnish excellent material for coal-tar-dye testing are candy, soft drinks, wine, tablets, jam, jelly, catsup, colored sugars, dessert powders, gelatin, etc.

Well-mounted pieces of wool dyed with these materials and placed in the notebook give it a living interest.

**Detection of Coal-Tar Dye in Butter.** Melt a quantity of butter the size of a marble in a test tube, being careful not to scorch it. Add an equal volume of Low's reagent and shake vigorously. Heat nearly to boiling and set aside. After the acid has settled it will be wine red in the presence of azo colors. Pure butter fat gives only a faint bluish tinge. (*Low's test.*)

*Low's Reagent.*  $\text{HC}_2\text{H}_3\text{O}_2$ , 4 parts;  $\text{H}_2\text{SO}_4$ , 1 part. Mix.

**Martin's Test.** Shake 5 g. of the butter in a test tube with 20 cc. of Martin's reagent. Let the mixture stand until the fat has settled to the bottom of the tube. The dye, if present, will color the supernatant liquid yellow.

*Martin's Reagent.*  $\text{CS}_2$ , 4 cc.;  $\text{C}_4\text{H}_5\text{OH}$ , 30 cc. Mix.

**Acid and Alkali Test.** Melt about 20 g. of the butter in an evaporating dish and set on a water bath until the curd and contained water have entirely separated. Pour off the clear, supernatant fat and filter it through a dry filter paper in a hot-water funnel or in an oven at  $60^\circ\text{C}$ . If the fat is not clear, it must be filtered again.

Pour into each of two test tubes 2 g. of this filtered fat dissolved in ether. Into one of the tubes pour 1 cc. of  $\text{HCl}$  and into the other the same volume of 10 per cent  $\text{KOH}$ . Shake the tubes well and allow to stand. In the presence of azo dye the test tube to which the acid has been added will show a pink to wine-red coloration, while the alkaline solution in the other tube will show no color. If annatto or other vegetable color has been used, the potash solution will be colored yellow (*Bulletin No. 107*).

**Coal-Tar Dye in Lemon Extract.** *Lythgoe's Test.* To the original sample add two or three drops of strong  $\text{HCl}$ .

No change indicates natural color, turmeric, or naphthol colors.

Pink indicates tropæolin or methyl orange.

Partial decoloration indicates Martin's yellow.

Complete decoloration indicates dinitro-cresols.

Evaporate 10 to 20 cc. of the sample to dryness, dissolve the residue in water, and employ the test of Sostegni and Carpentieri.

Why are flavoring extracts often artificially colored?

## SECTION XIII

### IDENTIFICATION OF VEGETABLE COLORS

**Caramel. Amthor's Test.** Place 10 cc. of the suspected solution in a Nessler tube or narrow clear-glass bottle. Add from 30 to 50 cc. of paraldehyde, the latter volume if the color is very dark. To make the solutions mix, introduce a few cubic centimeters of absolute alcohol.

If caramel is present, a yellow to dark-brown precipitate will fall to the bottom of the tube.

**Fuller's-Earth Method.** Shake 50 cc. of the solution with 25 g. of fuller's earth. Allow the mixture to stand in a corked bottle of about 2 in. diameter for an hour at room temperature.

The caramel will be absorbed by the earth, and the supernatant liquid will appear more or less clear, depending upon the amount of the absorption.

As some fuller's earth absorbs color more readily than others, it is advisable to experiment with different samples upon solutions known to contain caramel.

**Amyl-Alcohol Method.** Shake 5 cc. of the solution with 10 cc. of amyl alcohol in a small vial for a minute or so. Allow the liquids to separate.

If caramel is present, the upper layer will be decolorized to a greater or less extent. The lower layer will be colored in proportion to the amount of caramel in the solution.

Test vanilla extract, whisky, ginger ale, soft drinks, and the like for caramel.

**Cochineal.** *Girard and Dupré's Method.* If the sample is catsup, canned fruit, or of this nature, triturate in a mortar

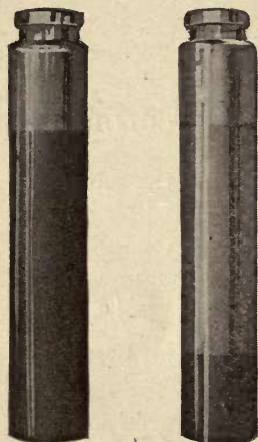


FIG. 17. True and artificial whisky treated with amyl alcohol

Caramel shows in the bottom of the right-hand bottle. The supernatant liquid is decolorized

with water until it is reduced to a very thin paste. Filter, acidulate with HCl, and shake with amyl alcohol. If cochineal is present, the alcohol will be colored yellow or orange. Separate the amyl alcohol and wash it with water until neutral. Add, drop by drop, a very dilute solution of uranium acetate. In the presence of cochineal a beautiful emerald-green color is produced.

**Turmeric.** Extract the color with alcohol. Saturate a filter paper with the extract and dry at  $100^{\circ}\text{C}$ . Dip the paper in a dilute solution of boric acid to which a few drops of 10 per cent HCl have been added. Dry the paper. Turmeric is indicated by a distinct cherry-red

coloration. Add a drop of alkali and olive-green will develop (*Bulletin No. 51, United States Bureau of Chemistry, p. 131*).

## SECTION XIV

### RAFFIA DYEING

Raffia is the cuticle of the leafstalks of the Madagascar palm (*Raffia ruffia*). Its tissue is cellulose for the most part, so the method of dyeing must necessarily differ from the method followed in dyeing wool, silk, or other animal fabrics.

A mordant is a substance to "set" a color; that is, to make the pigment unite chemically, or in some cases physically, or both, with the material dyed. The principal mordants used in raffia dyeing are alum and other aluminium salts, and certain salts of iron, tin, and copper.

Raffia is soaked in the mordant solution until thoroughly impregnated, and then boiled in a solution of the dye, which forms with the mordant a metallic colored substance known as a "lake." A lake is relatively insoluble and cannot be easily washed out.

**General Principles.** Alum should be used as a mordant unless otherwise specified. Dissolve 1/4 lb. in 10 qt. of water. Let the raffia stand in this solution until it has become thoroughly saturated. From six to twenty-four hours is generally sufficient. Always untie the bundles and do them up loosely.

Raffia should not be dry when placed in the dye. Either take it directly from the mordant or wet it thoroughly with water. Let the dye be boiling when the raffia is placed in it.

Alum spots, grayish patches on the finished product, are not generally disadvantageous. Most autumn foliage has a white fungus which, in general appearance, these spots resemble. A dip in warm water will remove them. Colors obtained as herein directed will compare favorably with the bright, soft colors of autumn or the fresh tints of spring.

Do not dye too dark. Lighter tints are more pleasing in woven and braided work. Dye slightly darker than desired, as the raffia is lighter when dried. Test pieces may be quickly dried by holding them against the side of the hot dye pan. This saves time and affords a convenient means of judging color value.

Strong dye and short boiling makes soft, flexible, tough raffia. Weak dye and long boiling makes harsh, brittle raffia. Some of the coal-tar dyes leave the material glossy, harsh, and brittle. Fifteen minutes should be the maximum time of boiling.

Vegetable dyes, in general, give soft, pleasing tones. Coal-tar dyes give bright, glossy colors.

Rinse before putting into a dye of another color. Dull, passive colors may be brightened by boiling in fresh or stronger dye. Weak dye is of little value. Keep the raffia well covered with dye, and turn frequently.

Dyes extracted from bark, leaves, fruit, roots, vegetables, wood, and the like should be carefully strained before using.

Save the waste dye, as many beautiful effects may be produced from it. It is not only economical to use this dye, but interesting to discover the different colors that may be obtained.

Time directions are only approximate, as much depends upon the quality and strength of the dye and on the kind and amount of mordant absorbed.



The outer side of the raffia will take a brighter tone than the inner side. This is especially noticeable in green and orange tones.

Do not place the full amount of raffia in the pan at once. Dye a small piece and see if the color suits you. Do not throw away the material which is displeasing in color, as it may easily be dyed black or brown.

Experiment by mixing small quantities of various dyes and mordants in cups and test tubes. You will doubtless discover some new and pleasing combination.

Material will absorb only a certain amount of the dye. This amount absorbed, the pigment point is reached. Do not expect to pour a quart of water at once into a pint cup.

Dye pans should be large enough for the work at hand. The four-quart or eight-quart size is convenient. The best results are obtained by using enamel ware.

**Preparation of Dyes and Mordants.** Dyes and mordants should be prepared in the following manner:

*Butternut.* Fill a four-quart pan half full of the husks; green ones give the softest tones; the shells are not objectionable. Cover with water and boil for fifteen minutes.

*Cardinal Red.* Dissolve a mass the size of a cranberry in a gallon of water.

*Copper Sulfate.* Two ounces to a quart of water.

*Elderberries.* Berries, 1 part; water, 3 parts.

*Fustic Chips.* A teacupful to 4 qt. of water. Boil ten minutes.

*Fustic Extract.* Dissolve a piece the size of a walnut in a gallon of water.

*Iron Sulfate.* Two ounces to a quart of water. Always use this solution in an old dish, as it will soon ruin a new one.

**Indigo.** Use indigo paste (sulfate of indigo). Dissolve a mass the size of two shoe buttons in 4 qt. of water.

**Logwood Chips.** See Fustic Chips.

**Logwood Extract.** See Fustic Extract.

**Leaves.** Fill a four-quart pan full of the shredded leaves well pressed down. Cover with water, and boil. Replenish the water from time to time until the dye appears highly colored. The usual time is about fifteen minutes.

**Onion Skins.** The outer skins from half a dozen medium-sized onions will furnish yellow dye for half a pound of raffia. Boil until the color is extracted. The time required is about ten minutes.

**Potassium Ferri-cyanide (Red Prussiate of Potash).** Two ounces to a quart of water.

**Potassium Dichromate.** Dissolve 1 oz. in a quart of water. This solution used with red dulls it and gives an orange tone.

**Sumac.** Three quarts of the broken cones. Keep well covered with water and boil for twenty minutes.

**Walnut.** See Butternut.

#### Specific Directions for obtaining the Following Colors :

**Black.** 1. Dye heavily with logwood and place immediately, without rinsing, into a strong, hot  $\text{FeSO}_4$  solution.

2. Dye as above, substituting oak leaves for the logwood.

**Blue.** Dye unmordanted raffia in indigo solution to which two or three drops of  $\text{H}_2\text{SO}_4$  have been added. Dry in direct sunlight. The brighter the light, the bluer the color. Many tones can be produced by dyeing for different lengths of time in this solution.

**Blue-Green.** 1. Boil mordanted raffia in plain indigo solution and dry away from the sunlight. Raffia dyed with indigo must be thoroughly rinsed to remove all traces of acid.

2. Dye in potassium ferri-cyanide to which a few drops of  $H_2SO_4$  have been added and mixed thoroughly.

3. Dye as in 2, and place directly into hot iron sulfate.

*Brown.* 1. Boil in dye from maple leaves until thoroughly colored. Remove, rinse, and wring out the superfluous liquid. Then boil in a dye prepared as follows: Strong butternut dye to which has been added 1/2 pt. of  $K_2Cr_2O_7$  solution and an equal amount of cardinal-red solution.

2. First dye in redwood solution and immerse for about three minutes in hot, strong logwood.

*Light Brown.* 1. Boil in clear butternut dye.

2. Boil in clear maple dye.

3. Dye green with fustic and indigo, q.v., and then boil in a solution of  $CuSO_4$ .

*Chocolate-Brown.* Dye in sumac and treat with  $FeSO_4$ .

*Dark-Brown.* Boil in butternut and then in  $K_2Cr_2O_7$ .

*Gray-Brown.* Equal amounts of sumac, maple, and oak leaves make a green-brown dye. Since red is the complement of green, and combined with it makes gray, add sufficient logwood to bring to the desired shade.

*Olive-Brown.* Boil for six minutes in dye from walnut husks.

*Red-Brown.* 1. Add a teaspoonful of alum to walnut dye and boil in the resulting solution for ten minutes.

2. Dye with cardinal and then with logwood.

3. (a) Dye in redwood solution.

(b) Then dye in a solution made from fustic chips, 3 parts, and logwood chips, 1 part.

*Yellow-Brown.* Boil first in a dye from maple leaves and then in  $K_2Cr_2O_7$ .

*Green. Bright Green.* Dye yellow with fustic and onion skins. Place immediately in a strong, hot solution of

indigo. Dull with iron sulfate to the desired shade. Nearly all tones can be produced by this method.

*Dull Green.* Dye with black birch leaves.

*Dark Green.* Dye with the birch and dip into  $\text{FeSO}_4$ .

*Gray-Green.* Add sufficient ammonia to elderberry juice to turn it a pronounced green. Boil the raffia in this solution until the desired shade is produced. The time required is about eight minutes.

*Olive-Green.* See Dark Green. Boil longer in the iron sulfate solution. If left too long, the raffia acquires a heavy olive tone not pleasing to the eye.

*Yellow-Green.* Color with fustic or onion skins and very slightly with indigo.

*Gray.* For these effects in general, boil in dye from sumac cones with the stems. A dip in iron sulfate will produce a pleasant neutral effect.

*Old Rose.* Dye with sumac berries, discarding all stems. Substitute elderberries for the sumac.

*Orange.* 1. Dye in fustic and then in strong, hot redwood infusion.

2. Boil in butternut for about six minutes. Rinse and dip into cardinal solution.

To produce a peculiar but pleasing effect, dye first in fustic and then in cardinal. One side will be brown yellow, the other a dark red.

*Red.* With infusions of redwood it is possible to get an almost complete color scale, from a deep, rich, dark red to a pale orange. These colors can be dulled with  $\text{FeSO}_4$ , thus producing an infinite variety of tints and shades.

For a deep red a very strong solution is needed, and only a small quantity of material can be colored at a time. As the strength is reduced the color tends toward orange.

The lighter tints of orange can be dulled with iron sulfate to give a pleasing light brown, practically identical with that produced from sumac.

*Bright Red.* Dye in sumac and strengthen in cardinal, or dye directly in the cardinal.

*Dark Red.* 1. Dye a bright red and dip in iron sulfate. The longer it remains the darker it becomes, until the limit is reached.

2. Dye brown with waste dye and then boil in cardinal.

*Indian Red.* 1. Dye orange red and boil in  $\text{CuSO}_4$  for two minutes.

2. Dye light brown and boil first in cardinal solution and then in potassium dichromate.

3. To butternut dye add half as much cardinal solution, one eighth as much  $\text{K}_2\text{Cr}_2\text{O}_7$ , one fourth teaspoonful of indigo paste, 2 oz. of logwood chips. Boil and strain, and dye the raffia in the clear liquid.

4. To produce a pale shade, dye in an extra strong solution from the sumac berries after discarding the stems.

*Purple-Red.* Dye in cardinal and then in indigo, or dye alum-mordanted raffia in logwood.

*Yellow.* Any of the yellow tones may be intensified by longer boiling.

*Lemon Yellow.* 1. Boil in dye from pear leaves to which a spoonful of alum has been added. Alum intensifies the color.

2. Boil for one minute in fustic, or for the same length of time in onion skins.

Any of the colors herein described can be duplicated, provided the experimenter becomes familiar with the special dye at hand.

Raffia dyeing is not merely a mechanical process; it is an art learned best by the patient and orderly worker.

## SECTION XV

### CHEMISTRY OF STAINS

A stain may be caused by the union, chemical or physical, or both, of some substance with a suitable medium, as paper, cloth, skin, and the like. The subject is a very deep and intricate one.

When a stain is purely physical, as, for example, a spot of grease on cloth, physical means of removing it are best; that is, some simple process of absorption or solution.

When the stain is of a chemical nature and certain oxids are formed, — take, for example, the stain of apple juice on cloth, — chemical means must be employed to reduce or “bleach” the oxid.

When a stain is of both a chemical and physical nature, both physical and chemical means should be employed to remove it. A stain of this character may be illustrated by an ink spot on cloth.

A good general bleaching reagent consists of a double solution kept in separate bottles.

No. 1. Acetic or tartaric acid solution, 20 per cent.

No. 2. Five grams of bleaching powder ( $\text{CaClO}_2$ ). Boil in 100 cc. of water until a pink color appears. Filter and add 50 cc. of cold water.

This combination is sometimes called “ink eradicator.” It must not be applied to silk or to fabrics of delicate color.

To remove stains of ink, coffee, tea, fruit, and dye, wet the spot thoroughly with No. 1. Absorb the superfluous liquid with a blotter and apply No. 2. Rinse and repeat,

if necessary. If a persistent, yellowish spot remains, as is often the case when woolen goods have been treated, remove all traces of the reagents and saturate with fresh  $H_2O_2$ . Common stains may be removed by treating as shown in the following table:

| STAIN   | REMOVED BY   |
|---|--|
| Acids . . . . .                                 | Cold water, Nos. 1 and 2   |
| Grass and fruit . . . . .                       | Cold water, alcohol, Nos. 1 and 2  |
| Grease . . . . .                                | Gasoline, carbon tetrachlorid, chloroform, ether, carbon bisulfid, ammonia, soapsuds, warm fuller's earth (cover with a blotter and apply a warm flatiron) |
| Dyes, coal-tar or of vegetable origin . . . . . | Nos. 1 and 2, ammonia  |
| Mildew . . . . .                                | Nos. 1 and 2, sunlight   |
| Inks . . . . .                                  | Nos. 1 and 2   |
| Inks, indelible (silver) . . . . .              | Potassium cyanide, 10 per cent. Use great caution, intensely <i>poisonous</i> . Sodium hyposulfite, 20 per cent  |
| Iodin . . . . .                                 | Methyl alcohol, potassium iodid solution, 10 per cent  |
| Iron rust. . . . .                              | Warm oxalic or citric acid, 10 per cent. If in silk, let it alone  |
| Paint, varnish . . . . .                        | Turpentine, benzine, carbon tetrachlorid. Use no turpentine on silk  |
| Tar, wagon grease . . . . .                     | Soap and oil, turpentine   |

## SECTION XVI

### FOOD PRESERVATIVES.

**Detection of Sulfurous Acid.** Weigh about 25 g. of the sample into a 200-cc. Erlenmeyer flask. Add water, if necessary, to form a thin paste and about 5 g. of sulfur-free zinc.

Introduce 10–20 cc. chemically pure HCl. Over the mouth of the flask place a small filter paper which has been wet with a strong solution of  $\text{Pb}(\text{NO}_3)_2$ . Heat gently. The blackening of the filter paper indicates the presence of sulfites. Why? A mere browning of the filter paper should not be accepted as evidence of the intentional addition of  $\text{SO}_2$ , either as a preservative or as a bleaching agent; it must be distinctly black. (*Bulletin No. 107, p. 187.*)

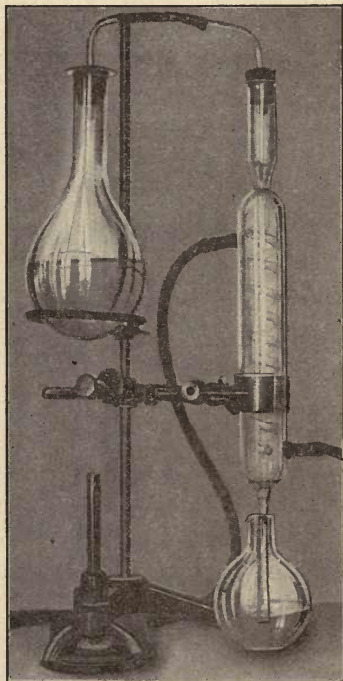


FIG. 18. Apparatus arranged for the detection of sulfurous acid by the distillation method

**Distillation Method.** *Leach.* Reduce 100–200 g. of the sample to paste as before, and acidify with 5 cc. of



20 per cent phosphoric acid. Transfer to a boiling flask and distil. Arrange the apparatus so that the outlet of the condenser will dip below the surface of a little water, about 20 cc.

Distil off 20 to 30 cc. Treat the distillate with 5 to 10 cc. of bromin water and boil for a minute or so.

Without waiting for the distillate to cool, add a little  $\text{BaCl}_2$ . A white precipitate indicates sulfurous acid.

What is this precipitate? Test its solubility. Of what use is the bromin water?

Test molasses, lime juice, mushrooms, Hamburg steak, sausage, etc. for sulfurous acid.

**Determination of Sulfurous Acid by Direct Titration.** Care must be taken in applying this method to other products than wine to determine whether the iodine is decolorized by any substance that may naturally be present.

Macerate 25 g. of the sample, if a solid or semisolid, with sufficient water to form a thin paste. Place in a 200-cc. Erlenmeyer flask. Add 25 cc. of normal KOH, mix thoroughly, and allow it to stand for fifteen minutes, shaking from time to time. Add 10 cc. of dilute sulfuric acid (1 to 3) and 5 cc. of freshly prepared starch solution. Rapidly titrate the mixture with N/50 iodine solution until a blue color is permanent for several minutes.

One cubic centimeter of N/50 iodine solution is equivalent to 0.00064 gram of sulfur dioxide.

From the per cent of  $\text{SO}_2$  calculate the per cent of sulfurous acid in the original sample (*Bulletin No. 107, p. 188*).

**Detection of Boron Compounds — Borax or Boric Acids.** It is not uncommon to find this forbidden preservative in cheese, ice-cream cones, fancy crackers and biscuits. It was formerly used in canned meats, but the practice has greatly declined of late.

"The common symptoms observed after long-continued doses of borax or boric acid in food are headaches, sensations of fullness in the head, uneasiness and nausea in the stomach, and disturbances of the digestion and appetite" (*Wiley*).

**Discover the Effects of  $H_3BO_3$  upon Turmeric Paper and Turmeric Tincture.** Break about 10 g. of saltines or other crackers into a crucible. Add a pinch of boric acid or borax, and ash. Acidulate the ash with a drop or so of HCl and dissolve in as little water as possible.

(a) Dip a strip of turmeric paper in the solution and allow it to dry. Result?

(b) Mix the remainder of the ash solution with a cubic centimeter of turmeric tincture in a watch glass and evaporate over a water bath. Result?

Confirm both (a) and (b) by placing a drop of dilute alkali upon the paper or on the contents of the glass. An olive-green color should appear.

After becoming familiar with the reaction between boric acid and turmeric, test crackers or biscuits, butter, cheese, canned meat, and shrimps for boron compounds.

**Method.** Ash about 10 g. of the sample, first adding enough limewater to make an alkaline reaction. Acidulate the ash with a drop or two of HCl. Dissolve in a few drops of water. Test with the turmeric paper and with the turmeric tincture, as outlined.

If the turmeric is reddened by the solution of the ash and turned olive-green by dilute alkali, boric acid, free or combined, is present in the sample.

**Boron Compounds in Butter.** Melt 25 g. of the sample on a water bath and allow the aqueous solution to settle. Decant this solution and acidulate with a drop or so of dilute HCl. From this point apply the regular turmeric test.

**Detection of Salicylic Acid ( $\text{HC}_7\text{H}_5\text{O}_3$ ).** This compound has been used for the preservation of catsup, jams and other fruit products, and beer. Reduce the sample to a thin paste with water, if it is not already a liquid. Acidify slightly with dilute  $\text{H}_2\text{SO}_4$ . Shake with an equal volume of chloroform in a closed flask or separatory funnel. Separate the chloroform and allow it to evaporate spontaneously.

*First Test.* To a part of the dry residue add a drop of ferric chlorid and an equal volume of water. A pronounced violet or purple color indicates the presence of salicylic acid.

*Second Test.* Heat the remainder of the residue gently with a few drops of 20 per cent  $\text{H}_2\text{SO}_4$  and a cubic centimeter of methyl alcohol.

If salicylic acid is present, a pronounced odor of "winter-green," or methyl salicylate, will be apparent.

**Detection of Benzoic Acid ( $\text{HC}_7\text{H}_5\text{O}_2$ ).** Benzoic acid is used for much the same purpose as salicylic acid, and is more often found in food products.

Extract the sample as for salicylic acid and evaporate the chloroform. Dissolve a part of the dried residue in ammonia and evaporate to dryness over a water bath.

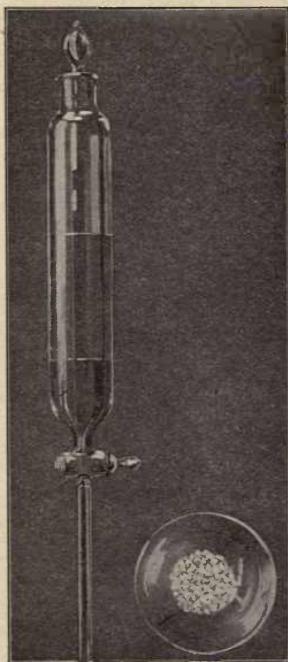


FIG. 19. Separatory funnel for extracting salicylic or benzoic acid with ether or chloroform

Sublimed crystals of benzoic acid show on the watch glass.

See Fig. 20

*First Test.* Dissolve in a few drops of water, heating gently to effect the solution. Filter into a small test tube and add a drop of ferric chlorid. A flesh-colored precipitate of ferric benzoate assures the presence of benzoic acid.



FIG. 20. Sand bath, filter paper, and double watch glass arranged to sublime benzoic acid

*Second Test.* Dissolve the remainder of the chloroform extract in ammonia and evaporate to dryness in a two-inch watch glass. Invert a second watch glass over the first. Between these insert a filter paper from the center of which has been cut a half-inch circle. Clamp the watch glasses closely together and heat at a low temperature on a sand bath. If benzoic acid is present, needlelike crystals will sublime on the upper watch glass. Examine them with a low-power lens. Dissolve and treat them with a drop of ferric chlorid, as in the preceding test.

**Detection of Saccharin.** Prepare the sample as for the salicylic acid test. Extract with ether and allow the latter to evaporate at room temperature. A distinctly sweet taste indicates the presence of saccharin.

Add a small piece of NaOH and heat gently. The saccharin will be converted into salicylic acid and can be detected by the ferric chlorid test.

## SECTION XVII

### EXAMINATION OF TOOTH POWDERS

The usual ingredients of these dentifrices are soap powder, precipitated chalk ( $\text{CaCO}_3$ ), sugar, orris root, and other flavoring materials.

Sometimes powdered pumice stone and cuttlefish bone are substituted for the chalk. Since these substitutes scratch the enamel, they are injurious and should be avoided. They may be detected as follows:

Shake up 1 to 2 g. of the powder with 10 cc. of dilute alcohol. To the residue add about 4 cc. of HCl and an equal volume of water. Note any effervescence. Boil. Allow any undissolved matter to settle and decant the solution. Insoluble matter indicates pumice stone. Confirm by placing a little on a glass plate and rubbing gently with a glass rod. If pumice is present, a scratching sound will be heard.

Divide the decanted solution into two parts.

*The First Part.* Evaporate to dryness and test with ammonium molybdate for the  $\text{PO}_4$  radical. If present, cuttlefish bone is indicated.

*The Second Part.* Test for calcium with  $(\text{NH}_4)_2\text{C}_2\text{O}_4$ . Its presence is indicated by a flocculent white precipitate. This further bears out the suggestion of the presence of powdered bone.

TABLE OF INDICATIONS

| EFFERVESCENCE | RESIDUE INSOL-<br>UBLE IN HCl | CALCIUM | PO <sub>4</sub> | INDICATION                    |
|---------------|-------------------------------|---------|-----------------|-------------------------------|
| Yes           | No                            | Yes     | No              | Chalk                         |
| Yes           | No                            | Yes     | Yes             | Cuttlefish bone               |
| No            | Yes                           | No      | No              | Pumice                        |
| Yes           | Yes                           | Yes     | No              | Chalk and pumice              |
| Yes           | Yes                           | Yes     | Yes             | Cuttlefish bone<br>and pumice |

What are the indications for chalk and cuttlefish bone?

In addition to the above tests, determine the free and combined alkali and the alkaline carbonates.

## SECTION XVIII

### EXPERIMENTS WITH GLUCOSE

Glucose is widely distributed in the vegetable kingdom. It occurs naturally in many fruits and vegetables, in honey, in the blood, liver, and urine. In the disease diabetes mellitus the quantity present in the urine is sometimes as high as 10 per cent.

It is artificially prepared on a large scale by treating corn or potato starch with dilute sulfuric acid. Its sweetness to that of sugar is as 3 to 5. Large quantities are annually consumed in the manufacture of leather, candy, table sirups, jams, jellies, and the like.

**Conversion of Starch into Glucose.** Boil 5 to 10 g. of sawdust, filter paper, cotton rags, or cornstarch, with a 10 per cent solution of  $H_2SO_4$  in an Erlenmeyer flask, in the neck of which is a funnel to act as a reflux condenser. Continue boiling until the liquid becomes a decided yellow or brown. Neutralize with powdered chalk and filter. Evaporate the filtrate to a thick sirup.

**Suggestions.** Pour the acid into the water.

Flasks will break if the starch is allowed to stick to the bottom. For the first attempt use corn or potato starch.

Replenish the water as it boils away. If the acid becomes too strong, it will carbonize the starch.

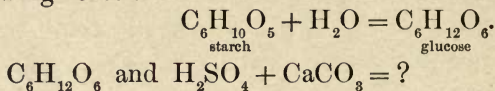
Neutralize by adding the chalk well powdered. Test often with litmus. Filter. If the filtrate is still acid, add

more of the carbonate. A relatively large quantity will probably be required.

Add plenty of water to the filter to wash the glucose from the spent carbonate.

Great care must be taken during the process of evaporation or the liquid will blacken. Stir constantly and finish over a water bath.

**Reactions.** Notice the interesting exhibition of catalysis in the following reaction :



Name the contents of the filter paper. Dry it. It will keep its form. Why? Which of the changes in the above experiment are physical, and which are chemical?

**Test for Glucose by the Reduction of a Copper Salt.** Dissolve a little glucose in water. Add 2 cc. Fehling's solution, 1 cc. each of No. 1 and No. 2. Heat nearly to boiling. The result is characteristic.

Repeat, substituting cane sugar for the glucose. Result?

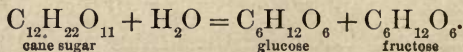
*Fehling's Solution, No. 1:*  $\text{CuSO}_4$ , 34.6 g.; water, 500 cc.

*Fehling's Solution, No. 2:* Rochelle salts, 173 g.; NaOH, 50 g.; water, 500 cc. Keep in separate bottles.

**Test for Glucose by the Precipitation of Dextrine.** Dissolve a little glucose in water. To two or three cubic centimeters add a large excess of methyl alcohol. Agitate. The glucose is precipitated as dextrine.

Repeat, substituting cane sugar for glucose. Result?

**Inversion of Cane Sugar.** Make a solution of cane sugar as before. Add one drop of HCl and boil vigorously. Test with Fehling's solution.





The mixture of glucose and fructose is called invert sugar. Notice how the inversion is brought about by hydrolysis. Before cane sugar is digested it must pass through the process of inversion.

Test honey, confectionery, maple sirup, molasses, jam, jelly, the contents of pies and cake fillers for invert sugar by the Fehling method.

Test the same for glucose by the precipitation of dextrine. It is instructive to treat a sample of homemade jelly and any one of the cheap varieties by this method. A decided milkiness in the alcohol indicates commercial glucose.

Many soft candies, waxes, taffies, a large proportion of stick candy, caramels, and the like are made with glucose. Sometimes a little cane sugar is added to give it a sweeter taste.

Considerable glucose is used in the manufacture of table sirups. These are sent to the market under euphonious names, as "Maple Drip," "Bon Ton," "Golden Drip," "White-Loaf Drip," etc.

**Detection of Glucose in Honey.** Dissolve one part of honey in an equal volume of water. Cool and add 5 to 8 drops of a dilute solution of iodine in KI.

If the honey solution remains a pale yellow, commercial glucose is probably absent. If decolorized, glucose is indicated.

If starch is present, the characteristic blue-to-purple coloration will appear (*Beckman's test*).

**Anilin-Acetate Test for Artificial Invert Sugar in Honey.** The reagent must be freshly prepared. Shake 5 cc. of chemically pure anilin with 5 cc. of water, and add 2 cc. of glacial acetic acid. The milky emulsion of anilin and water should clear up perfectly upon the addition of the acid.

Dissolve about 5 cc. of honey in a test tube with an equal volume of water, and pour a little of the anilin solution down the sides of the tube so as to form a thin layer upon the surface of the liquid. If artificial invert sugar is present, a red ring will form beneath this layer, and on gently agitating the tube all of the acetate will be tinged this color.

If the honey is pure and has not been overheated, no trace of the red will be found.

**Sugar in Vegetables and Fruits.** Grind a quarter of a turnip or half an apple through a food cutter. Place the pulp in a piece of cheesecloth and squeeze the juice into a beaker. Taste the juice. Pour about 5 cc. into a test tube and dilute with three or four times its volume of water. Test with Fehling's solution.

Test beets, carrots, parsnips, etc., for sugar. How does the sugar from the beet differ from that of the apple?

**Conversion of Starch to Invert Sugar by the Process of Mastication.** Grind a few grams of soda or milk crackers in a mortar with enough water to form a thin paste. Transfer to a test tube and test with Fehling's solution. Result?

Thoroughly chew about 5 g. of the cracker for about one minute. Test as before. How do you account for the change? Could the food be chewed too much? Why?

**Detection of Adulteration in Maple Sirup. Coloring Matter.** Shake 15 cc. of the sirup with 3 cc. of amyl alcohol and 1 cc. of  $H_3PO_4$  (20 per cent). Allow to settle. The amyl-alcohol layer should be a decided brown. Adulterated samples give a straw-to-light-brown color.

**Foam Test.** Mix 5 cc. of the sirup and 10 cc. of water in a graduated tube and shake vigorously for half a minute. Allow to stand ten minutes. The foam should not measure less than 3 cc. Adulterated samples give less foam.

**Precipitate Test.** Mix 5 cc. of the sirup with 50 cc. of water in a 50-cc. cylinder. Add 2 cc. of lead subacetate solution. Mix well and allow to stand for twenty hours. The precipitate should not measure less than 3 cc. (*Bulletin of Pharmacy, December, 1908.*)

When maple sugar is to be tested, dissolve 15 g. in enough water to make 15 cc. of sirup.

## SECTION XIX

### EXAMINATION OF HEADACHE POWDERS

A great deal of injury is done each year by the indiscriminate use of headache powders, "cures," "stops," tablets for car sickness, anti-pain tablets and pills, cold "cures," and the like.

Many of these contain a coal-tar derivative commonly known as acetanilid, or antifebrin. This is the acetyl derivative of anilin, and is therefore called phenylacetamid by the chemist. This substance is a dangerous heart depressant and should never be used except by the intelligent advice of a physician.

Another substance frequently used in cheap powders of the above description is phenacetin, known to the chemist as oxyethylacetanilid. If one values his health, he should avoid the common use of such pernicious drugs.

**Detection of Acetanilid in Headache Cures, Cold and Grippe Powders, and like Nostrums. *Strobel's Test.*** Place about 0.2 g. of the sample in a 5-in. test tube and add about the same volume of  $ZnCl_2$ . Heat gently, meanwhile holding a wood shaving or splint down the mouth of the tube. White fumes soon appear. Continue heating; the mixture melts, turns light yellow and finally black. Observe the shaving from time to time. If it is stained yellow, acetanilid is undoubtedly present. Note the peculiar odor of the fumes. Varnish or shellac the splint and mount it in the notebook with the label from the package.

**Ritsert's Test.** Boil 1 g. of the sample in a small beaker for two or three minutes with about 3 cc. strong HCl. Cool and divide into three portions and test in small tubes.

1. Add carefully 1 to 3 drops of a solution of bleaching powder ( $\text{CaClO}_x$ ), 1 to 200, in such a manner that the two liquids do not mix. A beautiful blue color is seen at the junction of the two liquids if acetanilid is present.

This is known as the indophenol reaction, and it responds to anilin compounds generally.

2. To another portion add a small drop of  $\text{KMnO}_4$ . A clear green color is formed if an appreciable amount of acetanilid is present.

3. Mix the third portion with a 3 per cent chromic acid solution. Acetanilid gives a green color, changing to a dark green in a few minutes, and forms a dark blue precipitate on the addition of a drop or two of NaOH solution.

In the case of powders containing vegetable matter or sugar of milk, both of which will turn brown on heating with HCl, it is advisable to first boil the sample in 5 to 10 cc. of water. Filter, cool the filtrate, and agitate. If acetanilid is present, it will crystallize out and settle. A centrifuge may be used to advantage to separate the crystals from the liquid. This latter which still contains some acetanilid in solution may be discarded and the crystals tested as indicated.

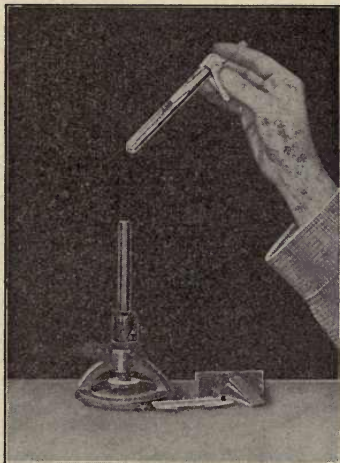


FIG. 21. Showing method of holding splint for the Strobel test

The blue color of the bleaching powder solution is probably due to the presence of anilin hydrochlorid.

Test headache powders for the bromin radical by gently heating with equal parts of  $\text{MnO}_2$  and  $\text{H}_2\text{SO}_4$ .

**Isonitрил Reaction.** Heat about a gram of the powder with 10 cc. of a 10 per cent NaOH solution. Remove from the flame and cautiously add a few drops of chloroform. Set aside for a few minutes. If acetanilid is present, the greasy, disgusting odor of phenylcarbamine will be apparent.

This last test is perhaps the best of all, and is especially applicable to a liquid or solid whose color might interfere with the Ritsert test.

**To determine the Per Cent of Acetanilid present.** Dissolve a definite weight of the powder, about 1 g., in hot water. Boil and filter. To the filtrate add bromin water until the yellow color persists. The acetanilid is precipitated as *p*-bromacetanilid. Dissolve the precipitate in benzol. Filter and evaporate the benzol over a water bath. Dry at a temperature not exceeding 100° F. Weigh the residue.

**Caution.** Keep the benzol from all flame, as it is exceedingly inflammable.

Pure acetanilid melts at 113° C. Determine the melting point of your sample.

If unfamiliar with any of the above tests, work with a known sample. Do not give up until you are familiar with them. You may save the life of some one.

## SECTION XX

### TESTS FOR ARSENIC

Arsenic is one of the most widely distributed elements. Unless care is taken by the manufacturer, it may contaminate our foods, articles of clothing, wall papers, paint, and the like. In testing any such substance for arsenic it is advisable to partially destroy the organic matter with a mixture of sulfuric and nitric acids. This treatment oxidizes the arsenic into arsenic acid, which may be completely removed with boiling water. The method advised is that of Chittenden and Donaldson (*Bulletin No. 86, Bureau of Chemistry, United States Department of Agriculture*).

To successfully perform tests for arsenic, it is absolutely necessary that all of the reagents are themselves free from it. Assure yourself of this first of all. Save time by putting a few scraps of arsenic-free zinc into four different flasks :

No. 1 containing 10 per cent HCl.

No. 2 containing 10 per cent  $H_2SO_4$ .

No. 3 containing 10 per cent nitro-sulfuric acid.

No. 4 containing 10 per cent HCl and a few drops of 10 per cent  $CuCl_2$ .

Over the mouth of each flask place a piece of filter paper, which has been wet with a few drops of concentrated solution of mercuric chlorid. There should be a brisk evolution of gas.

If after half an hour the filter paper shows no discoloration, the reagents are suitable for use.

*Preparation of the Sample. Wall Paper, Cloth, etc.* Place about 25 sq. cm. of the sample cut into small pieces in an evaporating dish. Treat with 1 to 5 cc. of a mixture of concentrated sulfuric and nitric acids, 30 to 1, both of which have been proved free from arsenic. Add a few drops of

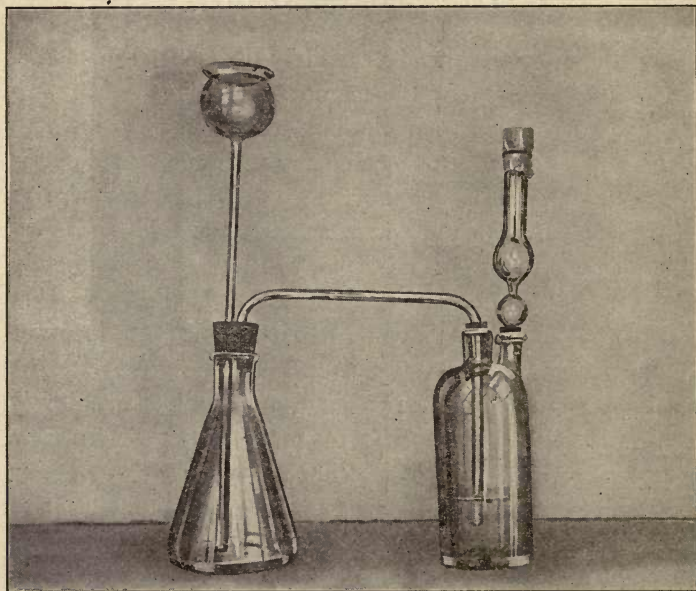


FIG. 22. Gutzeit apparatus for the detection of arsenic

Showing wash bottle with outlet capped with prepared filter paper

water and allow the action to proceed for five minutes. Heat with a low flame until all of the acid is driven off, or until the residue has granulated and the fumes have nearly disappeared. Break up the charred mass, add a little water, and boil to get rid of the  $\text{H}_2\text{SO}_3$ . Filter through a small filter and wash to about 40 cc.



**Preparation of the Sample.** *Meats, Vegetables, etc.* Heat in a porcelain dish about 100 g. of the sample with 23 cc. of  $\text{HNO}_3$ , stirring occasionally with a glass rod. When the substance has become a deep yellow or orange color, remove from the heat and add 3 cc. of  $\text{H}_2\text{SO}_4$ . Stir the contents of the dish while the nitrous fumes are given off. Care should be taken to protect the hands from these fumes.

Heat gently and add while hot, drop by drop, 8 cc. of  $\text{HNO}_3$ , stirring the mass constantly. Heat more strongly until acid fumes come off and a charred mixture remains. Break this up, extract with boiling water, and filter as in the case of wall paper.

**NOTE.** Always conduct these preliminary processes under a gas hood or out of doors.

**The Gutzeit Test.** Into a clean Erlenmeyer flask of about 200 cc. capacity, fitted with a thistle tube and a right-angled delivery tube, place a few pieces of arsenic-free zinc. Slide the zinc gently into the flask to avoid breakage. Pour in the filtrate from the prepared sample and about 5 cc. of the tested  $\text{HCl}$ , containing half a cubic centimeter of a 10 per cent solution of  $\text{CuCl}_2$  of known purity.

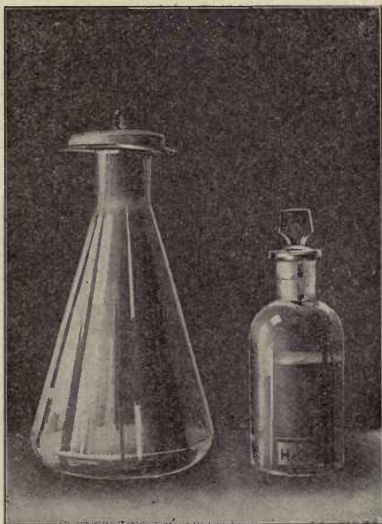


FIG. 23. Simple form of Gutzeit apparatus

Showing the prepared filter paper held over the mouth of the flask

Allow the escaping gas to pass through a few cubic centimeters of lead acetate solution contained in a small wash bottle or potash bulb, and impinge upon a piece of Swedish filter paper which has been wet with a drop or so of a concentrated solution of mercuric chlorid.

If after half an hour the paper shows a stain yellow to deep orange, arsenic is present. The color varies with the amount. Very large quantities produce a yellowish-brown stain.

Write the reaction between free hydrogen and arsenic. What compound causes the yellow color on the filter paper? If the filter paper remains white, freedom from arsenic is assured.

Place a little chemically pure 10 per cent HCl upon perfectly pure zinc. Result? Then add a few drops of  $\text{CuCl}_2$  solution. How do you explain the effect? Why was the copper chlorid solution added to the original sample?

Make arsenic tests upon the following: wrapping paper, samples of cloth, candy wrappings, wall paper, etc. Liquids can usually be tested without subjecting them to the previous preparation.

## SECTION XXI

### METHOD FOR TESTING PAINT AND OILS

If one desires to paint his building with a lead and zinc paint and pays the price, he should get lead and zinc, not sand, lime, and barium sulfate or barytes. On the other hand, if he pays a sand-and-lime price, he should not expect to get a metallic article. Which kind have you seen used?

**Extraction of the Oil.** Place about 5 g. of the paint in a small flask or beaker and wash with successive portions of warm petroleum ether, or benzine. Heat this in a water bath, away from any flame. Continue the washing until a few drops leave no residue on evaporation.

It is more convenient to conduct the extraction with a Soxhlet apparatus, placing the paint in an extraction shell the weight of which must be previously determined. Dry the undissolved residue at 100° C. and calculate the per cent of the oil.

**To test the Purity of the Oil.** Evaporate the ether extract from the previous experiment. Warm 2 cc. of the oil so obtained in a test tube and add an equal volume of glacial acetic acid. Cool in running water and add one drop of concentrated  $H_2SO_4$ . Pure linseed oil turns sea-green, the color deepening on standing. A fugitive violet color indicates rosin oil.

Heat a little of the oil in a test tube to about 100° C. Cool and rub on the back of the hand. If present, fish oil will be detected by the characteristic fishy odor.

**To test the Body of the Paint.** 1. Boil a portion of the residue with strong acetic acid. A residue indicates  $\text{BaSO}_4$  or sand ( $\text{SiO}_2$ ), or both. Save the filtrate.

2. To a second portion add an equal volume of  $\text{Na}_2\text{CO}_3$ , then mix and fuse with a blowpipe on charcoal. If lead is present, a small metallic globule will fuse out. Lead in paint is usually present in the form of basic lead carbonate,  $2\text{PbCO}_3 \cdot \text{Pb}(\text{OH})_2$ .

3. Moisten a third portion with a solution of  $\text{Co}(\text{NO}_3)_2$ . Heat strongly as before. A green color indicates zinc. Probably  $\text{ZnO}$  is in the original paint.

4. To the filtrate from 1 add an excess of  $\text{NH}_4\text{OH}$  and then ammonium oxalate. If lime is present, a white precipitate of oxalate of lime is formed.

Suggest a method for determining how much of each ingredient is present.

**Test for the Purity of Olive Oil.** Shake equal volumes (5 cc.) of the oil and  $\text{HNO}_3$ . Pure oil should turn from pale to dark green in a few minutes. If it changes to brown, red, or orange, the addition of a foreign oil is indicated.

Heat for five minutes in a water bath at  $100^\circ\text{C}$ . It should become pale yellow to orange yellow. On standing it will, if pure, become a yellow solid. (*Leach's test.*)

**Free Fatty Acids in Olive Oil.** Weigh exactly 20 g. of the sample into a counterpoised Erlenmeyer flask and add 50 cc. of neutral alcohol. Mix thoroughly and heat the contents to  $60^\circ\text{C}$ .

Titrate with tenth-normal sodium hydrate, using phenolphthalein as an indicator. Shake the mixture often during the titration. The number of cubic centimeters of the alkali used to neutralize the acid in 1 g. of the oil is called the Acid Number.

Each cubic centimeter of tenth-normal sodium hydrate equals 0.0282 g. of oleic acid. Tabulate the results as Acid Number and also as Per Cent of Oleic Acid. ~

The fresher and better grades of olive oils contain the least amount of free fatty acid.

*Neutral Alcohol.* Titrate any convenient volume of 95 per cent alcohol with tenth-normal alkali, using phenolphthalein as an indicator.

BACH'S TABLE FOR OIL REACTIONS

| KIND OF OIL      | AFTER AGITATION WITH HNO <sub>3</sub> | AFTER HEATING FOR FIVE MINUTES ON WATER BATH | CONSISTENCY AFTER STANDING TWELVE TO EIGHTEEN HOURS |
|------------------|---------------------------------------|--|---|
| Olive . . . .    | Pale green                            | Orange to yellow                             | Solid   |
| Peanut . . . .   | Pale rose                             | Brownish yellow                              | Solid   |
| Rape . . . .     | Pale rose                             | Orange yellow                                | Solid   |
| Sesame . . . .   | White                                 | Brownish yellow                              | Liquid  |
| Sunflower . . .  | Dirty white                           | Reddish yellow                               | Buttery   |
| Cottonseed . . . | Yellowish brown                       | Reddish brown                                | Buttery   |
| Castor . . . .   | Pale rose                             | Golden yellow                                | Buttery   |

**Detection of Cottonseed Oil in Olive Oil.** Take any convenient volume of CS<sub>2</sub> in which 1 per cent of sulfur has been previously dissolved, and add an equal volume of amyl alcohol (fusel oil).

To 5 cc. of the sample add an equal volume of the reagent. Stop the test tube loosely with cotton and heat for fifteen minutes in a bath of saturated boiling brine.

If cottonseed oil is present, a deep red to orange color is developed. Pure olive oil gives little or no color under this treatment. (*Halphen test.*)

This test can also be used for the detection of cottonseed oil in lard. The fat from animals fed on cottonseed meal is said to give a faint reaction.

If the olive oil containing cottonseed oil has been previously heated, the reaction is much less delicate.

Kapok oil, from the seeds of the *Eriodendron anfractuosum*, and baobab oil give the same reaction. A distinction can, however, be made, since the two last oils react without heating, while cottonseed oil must be heated.

**To distinguish Renovated from Creamery or Dairy Butter.**

Melt two or three grams of butter in an iron spoon. Pure butter melts quietly with the production of much foam. Renovated butter and oleomargarine bump and sputter like hot grease and produce no foam to speak of.

**Waterhouse Test for Oleomargarine.** Thoroughly shake 50 cc. of sweet milk and heat nearly to boiling. Add from 2 to 5 g. of the sample and stir with a small wooden stick flattened at one end until the fat is entirely melted.

Place the beaker in a dish of ice water and continue stirring until the fat solidifies.

If the sample is oleomargarine, the fat can be collected into a lump. Butter fat cannot be so collected, but is more or less emulsified with the milk.

If the sample is renovated butter, it will tend to collect as a film on the surface of the milk when the stirring is stopped. It does not clot or gather like oleomargarine, but usually adheres to the wooden rod.

## SECTION XXII

### DETERMINATION OF FOOD VALUES

**Total Nitrogen and Proteids of Cereal Products.** *Gunning's Method.* This method consists in decomposing the organic matter by prolonged digestion with sulfuric acid and potassium sulfate. The carbon is driven off as  $\text{CO}_2$  and the hydrogen as water. The nitrogen is converted into ammonium sulfate from which the free ammonia is liberated by means of an alkali and distilled into a known volume of N/10 acid, and the amount calculated by titrating the acid remaining.

It should be observed that foods in their natural state seldom if ever contain nitrates. Should this radical be present in appreciable amounts, the Gunning method must be modified.

Prove the absence of nitrates by extracting about 5 g. of the sample with water. Filter and test the filtrate by mixing with a solution of ferrous sulfate. Add concentrated sulfuric acid so as to form a layer below the mixed solution. In the presence of nitrates a dark brown ring forms at the juncture of the two liquids.

Prove the reliability of this reaction by working with a solution known to contain a nitrate.

In the absence of nitrates proceed with the Gunning method as follows:

Weigh exactly 0.5 g. of the finely powdered sample — bread, macaroni, breakfast food, etc. — and transfer to a

clean, dry Kjeldahl flask of about 250 cc. volume. Add 10 g. of  $K_2SO_4$  and from 15 to 25 cc. concentrated  $H_2SO_4$ . Incline the flask at an angle of about  $75^\circ$  over a small bare flame and heat gently until all foaming stops.

The mixture is now of a dark brown color. Slip a piece of wire gauze under the flask and slightly increase the heat

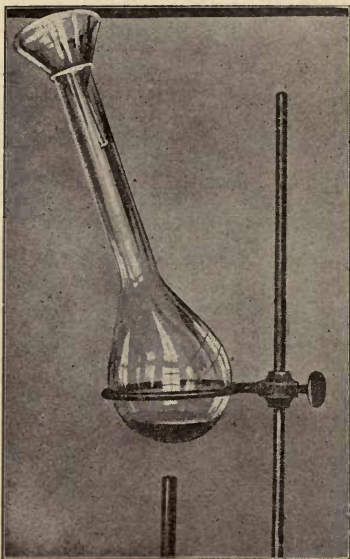


FIG. 24. Kjeldahl flask arranged for the determination of nitrogen

until the gauze is cherry red where it comes in contact with the bottom of the flask.

Place a funnel in the neck of the flask to act as a reflux condenser. Continue heating until the contents are colorless or of a pale straw color. This usually takes from thirty minutes to two hours. Conduct the heating in a gas hood.

Allow the flask to cool. Transfer the liquid into a boiling flask of about 500 cc. capacity, rinsing carefully with 200 cc. of water.

Add sufficient saturated solution of  $NaOH$  to make

the contents strongly alkaline, using phenolphthalein as an indicator.

Place two or three pieces of zinc in the boiling flask to prevent bumping, and distil off at least 150 cc., using a vertical condenser whose outlet dips below the surface of exactly 50 cc. of  $N/10 H_2SO_4$  contained in the receiving flask. (See apparatus for the detection of sulfurous acid.)



It is of the utmost importance to know the exact volume of standard acid in the receiving flask. Measure with an accurate pipette.

When all the ammonia has been distilled and absorbed, titrate the contents of the receiving flask with N/10 NaOH, using cochineal as an indicator.

The difference between the original volume of the standard acid and the volume of N/10 NaOH required to titrate it represents the number of cubic centimeters of N/10 H<sub>2</sub>SO<sub>4</sub>

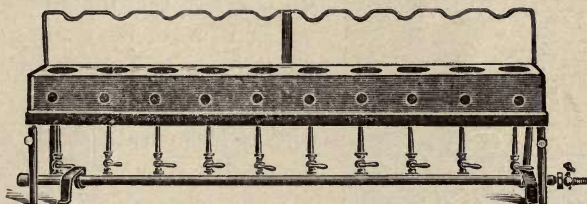


FIG. 25. Digesting shelf for making simultaneous nitrogen determinations

neutralized by the liberated ammonia. Every cubic centimeter of N/10 sulfuric acid represents .0014 g. of nitrogen.

The proteids are calculated from the total nitrogen by multiplying by the factor 6.25. This factor is the one generally adopted in determinations of this kind.

EXAMPLE. Weight of cereal = 0.5 g.

Volume of N/10 H<sub>2</sub>SO<sub>4</sub> = 50 cc.

Required 44 cc. of N/10 NaOH to titrate the excess acid.

Therefore 6 cc. of N/10 H<sub>2</sub>SO<sub>4</sub> were neutralized by the liberated ammonia.

1 cc. of N/10 H<sub>2</sub>SO<sub>4</sub> = .0014 g. of nitrogen.

6 cc. of N/10 H<sub>2</sub>SO<sub>4</sub> = 6 × .0014, or .0084 g. of nitrogen.

6.25 × .0084 = .0525 g. of proteid.

.0525/.5 = 10.5 per cent of proteid.

In such an analysis as the preceding one it is customary to state that the Protein = 6.25 × N.

**Fat of Cereal Products, known as "Ether Extract."** Weigh from 2 to 3 g. of the sample into a tared extraction shell (*Schleicher and Schull*). Dry thoroughly at  $212^{\circ}$ .

Place the shell in a Soxhlet or Wiley Extractor and extract with water-free ether. Dry the shell and residue to constant weight and by difference calculate the ether-soluble matter.

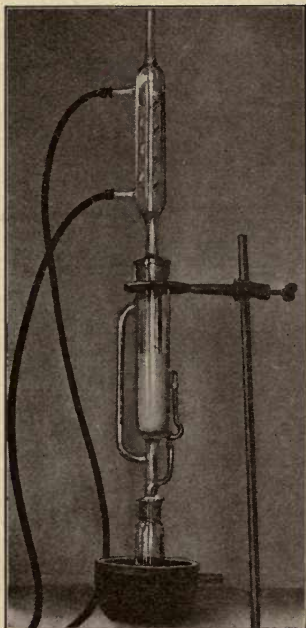


FIG. 26. Soxhlet extraction apparatus properly set up

A thorough extraction requires several hours. Great caution must be exercised in heating the extracting apparatus lest the ether take fire. Use a large water bath and a small flame, or, still better, an electric stove.

**Water or Moisture in Cereal Products.** Weigh from 2 to 5 g. of the sample into a tared watch glass. Spread it evenly over the bottom, forming as thin a layer as possible. Dry at  $100^{\circ}\text{C}$ ., cool, and reweigh. Calculate the per cent of water by difference.

**Ash or Mineral Content of Cereal Products.** Transfer the dried residue from the water determination to a tared porcelain crucible, taking care that none of the sample is lost. Burn to a white ash at the lowest temperature possible. If too much heat is employed, the ash will fuse to the bottom of the crucible. Cool in a desiccator, reweigh, and calculate the per cent.

**Carbohydrates in Cereal Products.** The carbohydrates are often expressed by adding the per cent of water, ash, proteids, and fat, and subtracting the sum from 100.

**Calculation of Fuel Value.** This value may be approximately determined by means of the Rubner factors, which give for each pound of protein or carbohydrate 1860 calories, and for each pound of fat 4220 calories.

**EXAMPLE.** Suppose the analysis of a certain cereal product shows : protein, 13.4 per cent ; carbohydrates, 74.1 per cent ; fat, 0.9 per cent.

|       |                               |                |          |
|-------|-------------------------------|----------------|----------|
| Then  | $1860 \times (.134 + .741) =$ | 1627.50        | calories |
| •     | $4220 \times .009 =$          | 37.98          | calories |
| Total |                               | <u>1665.48</u> |          |

## SECTION XXIII

### TESTING URINE<sup>1</sup>

**Determine Reaction.** Normal, slightly acid; after a full meal may be alkaline.

**Determine Odor.** Normal, peculiar, aromatic.

**Determine Color.** Normal, pale straw to reddish yellow. May be very pale by nervousness or excessive drinking.

**Determine Specific Gravity.** Normal, 1.015 to 1.025 at 60° F.

**Determine Total Solids.** Normal, 3.4 per cent to 5.8 per cent. Total solids equal (specific gravity - 1) multiplied by 2.33. This is equivalent to the number of grams per cubic centimeter.

**To detect Albumen and Phosphates.** *First Method.* Fill a test tube half full of clear urine. Boil the upper portion of the liquid. A turbidity indicates <sup>phosphates</sup> albumen or  $\text{PO}_4$ , or both. Add a drop of acetic or nitric acid; the phosphates dissolve, the albumen does not.

*Second Method.* Place about a cubic centimeter of concentrated  $\text{HNO}_3$  in a test tube, and by means of a pipette allow two or three cubic centimeters of the urine to rest upon its surface.

If albumen is present, a white zone or flocculent precipitate forms at the ring of contact of the two liquids. The extent of turbidity indicates roughly the amount of albumen present.

<sup>1</sup> For more complete analysis see Merck's *Manual* for 1911.

A green turbidity indicates biliary pigments. Reddish brown indicates blood.

**Urates or Uric Acid.** *Murexide Test.* Evaporate a few drops of urine to dryness on a watch glass. Add a drop or two of  $\text{HNO}_3$  and again cautiously evaporate. Then add an equal volume of  $\text{NH}_4\text{OH}$ . A purple color indicates urates, uric acid, or both.

**Invert Sugar.** *Fehling's Test.* Boil 5 cc. of Fehling's solution, and if the color does not change, add an equal volume (not more) of urine, and boil. In the presence of reducing sugars the characteristic red-to-yellow precipitate forms. (See tests for glucose.) Use this test only when uric acid is absent.

*Haines's Test.* Reagents:  $\text{CuSO}_4$ , 2 g.; glycerin, 20 g.;  $\text{KOH}$ , 9 g.; water, 175 g. Boil 4 cc. of the solution and add 6 to 10 drops (not more) of the urine and boil again. In the presence of reducing sugars the yellow-to-red precipitate forms.

**Detection of Sugar in the Presence of Urates or of Uric Acid.** Heat 1 g. of phenylhydrazine hydrochlorate, 2 g. of sodium acetate, and 25 cc. of urine, and if the salts do not completely dissolve, add a little water, and place in boiling water.

Remove after twenty minutes to cold water. If sugar is present, characteristic crystals of phenylglucosazone form.

**Chlorids.** Add a few drops of nitric acid to the urine to prevent the precipitation of the phosphates, and gradually add a few drops of  $\text{AgNO}_3$ . A white precipitate soluble in ammonia indicates chlorids. If present in small quantity, a milky color only will be seen.

**Sulfates.** Use  $\text{BaCl}_2$  instead of the silver nitrate. If present, the insoluble precipitate of  $\text{BaSO}_4$  will be seen.

## SECTION XXIV

### SELECTED EXERCISES

**An Experiment with the Albumen of Meat.** The most important solid constituent of the body of an animal is albumen. Place about 20 g. of lean beef finely minced in a beaker of cold water and gradually heat to about 130° F. Remove, filter the liquid, and test as follows:

To a portion add  $\text{HNO}_3$ . A white precipitate or a decided milkiness indicates albumen.

To another portion add a few drops of iodine. A yellow or port-wine color indicates the presence of glycogen or animal starch.

Repeat the above experiment by placing the same weight of beef in actively boiling water. Leave for a minute and test as before. How do you account for the difference? What does this show concerning the cooking of meats?

**Examination of Common Salt. Moisture.** Purchase as many different brands of table salt as possible; also several samples of "coarse-fine" and rock salt.

Place exactly 5 g. of the sample in a small tared Erlenmeyer flask and heat to a temperature not exceeding 150° C. for three hours on a sand bath. Remove from the bath, insert a funnel in the mouth of the flask, and allow the contents to cool. The introduction of the funnel renders the use of a desiccator unnecessary for this determination. Reweigh, and from the loss of weight calculate the per cent of moisture. Reserve the residue for the determination of  $\text{MgCl}_2$ .

**Insoluble Matter.** Dissolve 5 g. of the sample in 100 cc. of water, heating gently if necessary. Filter the solution through a balanced filter paper, washing the residue with warm water until the filtrate shows no precipitate with  $\text{AgNO}_3$  solution. Dry and weigh the contents of the filter paper and calculate the per cent of insoluble matter.

**Chlorin.** Dissolve 5 g. of the undried sample in a little water and make up the solution to exactly 500 cc. in a measuring flask. Mix thoroughly and withdraw 10 cc. by means of a pipette. Place in a clean beaker and add an equal volume of distilled water. Titrate with N/10  $\text{AgNO}_3$ , using neutral potassium chromate as an indicator (see p. 30). A liter of N/10 silver nitrate contains 17 g. of the pure crystallized salt.

Deduct 0.1 cc. of the silver solution added, as this amount is required to produce the permanent red tinge. Every cubic centimeter of the N/10  $\text{AgNO}_3$  is equivalent to 0.00355 g. of chlorin.

The 10 cc. of the titrated salt solution contained, theoretically, how many grams of chlorin? How many grams did you find? What was the per cent of chlorin?

**Suggestion.** How many grams of salt did you dissolve? To what volume did you dilute it? How many grams of salt in 10 cc. of this solution?

**Calcium Sulfate. First Method.** Dissolve 5 g. of the sample in 20 cc. of water to which 2 cc. of HCl have been added. Boil gently, being careful to lose none of the solution during the process. In the case of rock salt it may be necessary to continue the treatment for some time in order to dissolve all of the  $\text{CaSO}_4$ .

Neutralize the solution with ammonia and precipitate the calcium with  $(\text{NH}_4)_2\text{C}_2\text{O}_4$ . Allow it to stand overnight

and filter the solution through a fine, ashless filter paper. Wash the residue carefully, dry, and ignite it in a weighed crucible until the oxalate is converted into CaO. This will require about twenty minutes at a white heat. Cool and weigh as CaO. One part of CaO is equivalent to 2.4271 parts of  $\text{CaSO}_4$ . Calculate the per cent of  $\text{CaSO}_4$  in the original sample.

*Second Method.* Dissolve 10 g. of salt in warm water containing 1 per cent of HCl. Dilute to a liter and draw out 250 cc. (2.5 g. of salt). Heat this portion to boiling, add 1 cc. of HCl, and immediately pour in about 20 cc. of boiling 10 per cent  $\text{BaCl}_2$ . Do not add the barium chlorid solution drop by drop, but introduce it all at once. The precipitate should settle in half an hour. Decant the clear portion through an ashless filter paper. Pour 100 cc. of boiling water on the precipitate, agitate, and allow to settle, which it should do in about four minutes. Decant again and repeat the operation until the liquid ceases to give an acid reaction. Finally wash the precipitate on the filter. Dry, ignite at a low heat, and weigh the  $\text{BaSO}_4$ . From this calculate the per cent of  $\text{CaSO}_4$  in the sample.

**Magnesium Chlorid.** Into the flask containing the dried residue from the moisture determination place 25 cc. of absolute alcohol. Cork the flask and gently shake the contents from time to time for ten minutes. Filter and evaporate the alcohol, which contains nothing but  $\text{MgCl}_2$ . Dissolve the residue in water and titrate with N/10  $\text{AgNO}_3$ .

From the chlorin found calculate the per cent of  $\text{MgCl}_2$  in the sample.



SYMBOLS, ATOMIC WEIGHTS, AND VALENCE OF THE MORE IMPORTANT ELEMENTS

| ELEMENT              | SYMBOL | ATOMIC WEIGHT | VALENCE |
|----------------------|--------|---------------|---------|
| Aluminium . . . . .  | Al     | 27.1          | 3       |
| Antimony . . . . .   | Sb     | 120.2         | 3, 5    |
| Arsenic . . . . .    | As     | 75            | 3, 5    |
| Barium . . . . .     | Ba     | 137.3         | 2       |
| Bismuth . . . . .    | Bi     | 208           | 3, 5    |
| Boron . . . . .      | B      | 11            | 3       |
| Bromin . . . . .     | Br     | 79.9          | 1       |
| Cadmium . . . . .    | Cd     | 112.4         | 2       |
| Calcium . . . . .    | Ca     | 40            | 2       |
| Carbon . . . . .     | C      | 12            | 4       |
| Chlorin . . . . .    | Cl     | 35.5          | 1       |
| Chromium . . . . .   | Cr     | 52.1          | 2, 3, 6 |
| Cobalt . . . . .     | Co     | 58.9          | 2       |
| Copper . . . . .     | Cu     | 63.6          | 1, 2    |
| Fluorin . . . . .    | F      | 19            | 1       |
| Gold . . . . .       | Au     | 197.2         | 1, 3    |
| Hydrogen . . . . .   | H      | 1             | 1       |
| Iodin . . . . .      | I      | 126.9         | 1       |
| Iron . . . . .       | Fe     | 55.8          | 2, 3    |
| Lead . . . . .       | Pb     | 207.1         | 2, 4    |
| Magnesium . . . . .  | Mg     | 24.3          | 2       |
| Manganese . . . . .  | Mn     | 54.9          | 2, 4    |
| Mercury . . . . .    | Hg     | 200           | 1, 2    |
| Nickel . . . . .     | Ni     | 58.6          | 2       |
| Nitrogen . . . . .   | N      | 14            | 3, 5    |
| Oxygen . . . . .     | O      | 16            | 2       |
| Phosphorus . . . . . | P      | 31            | 3, 5    |
| Platinum . . . . .   | Pt     | 195           | 4       |
| Potassium . . . . .  | K      | 39.1          | 1       |
| Silicon . . . . .    | Si     | 28.3          | 4       |
| Silver . . . . .     | Ag     | 107.8         | 1       |
| Sodium . . . . .     | Na     | 23            | 1       |
| Strontium . . . . .  | Sr     | 87.6          | 2       |
| Sulfur . . . . .     | S      | 32            | 2, 4, 6 |
| Tin . . . . .        | Sn     | 119           | 2, 4    |
| Zinc . . . . .       | Zn     | 65.3          | 2       |



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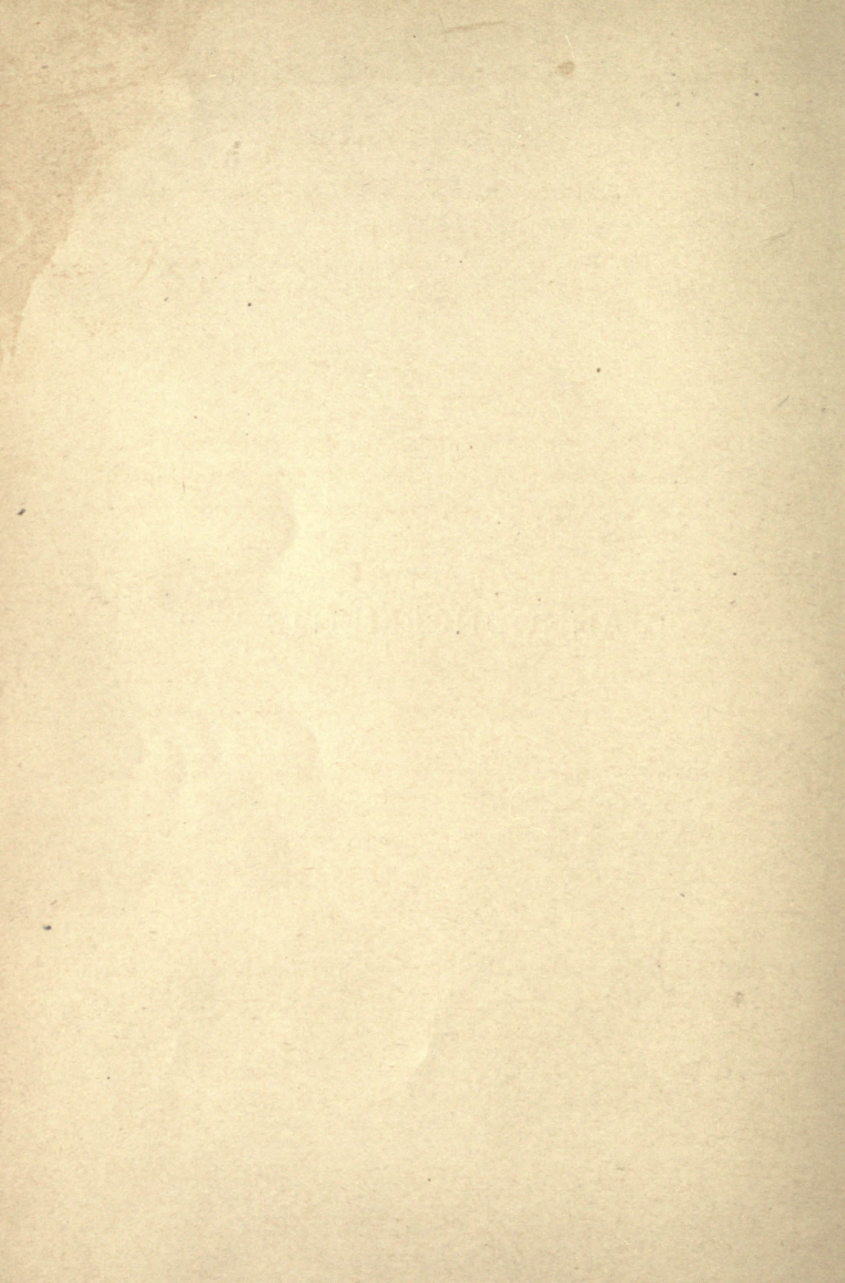
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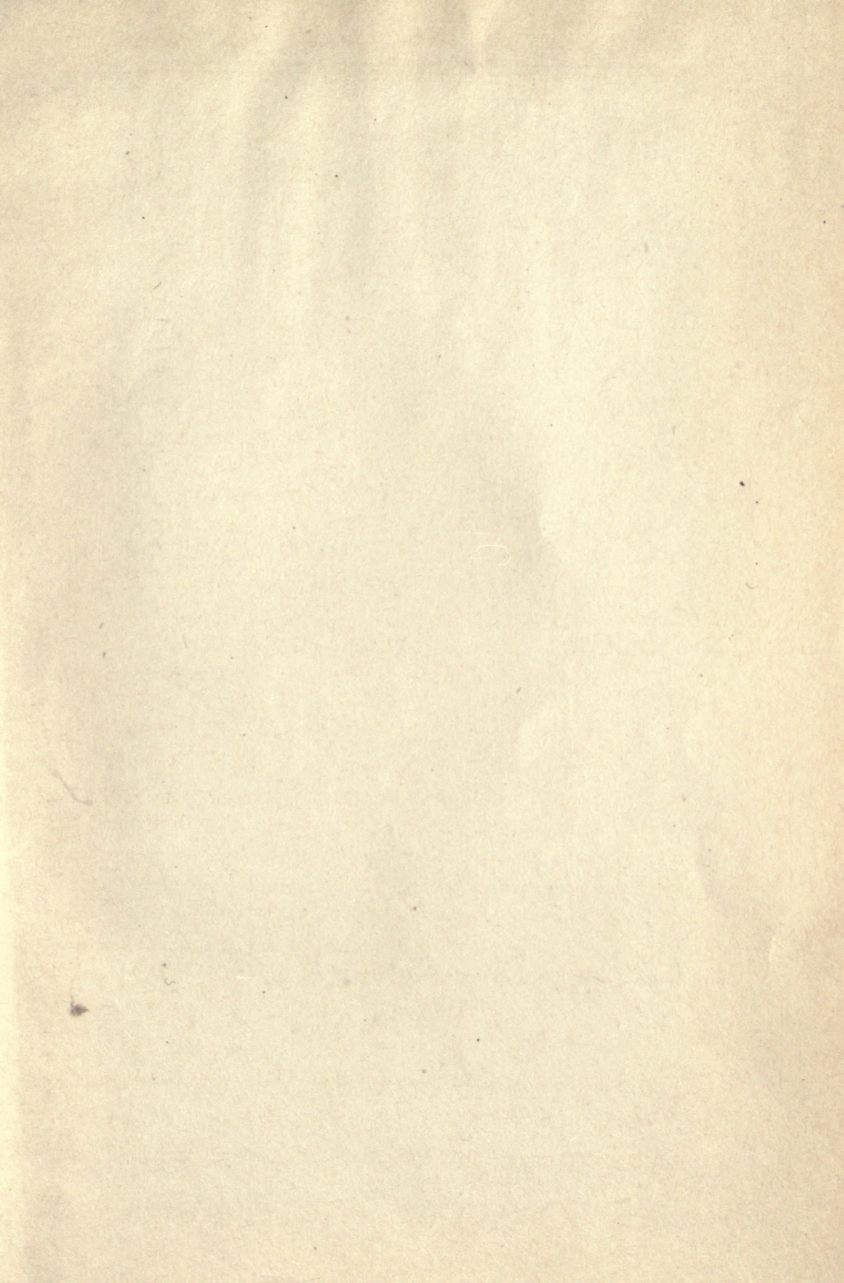
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