# HOUSEHOLD Chemistry



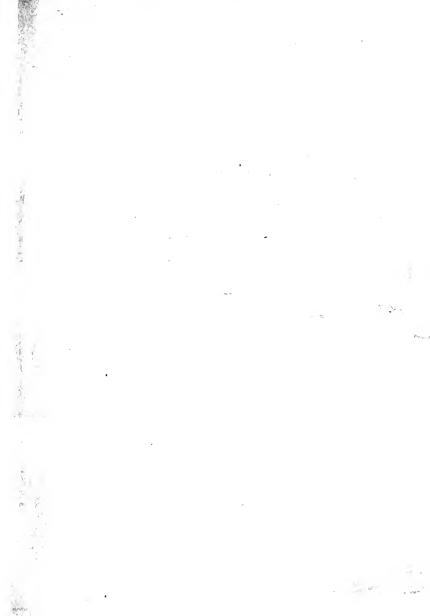
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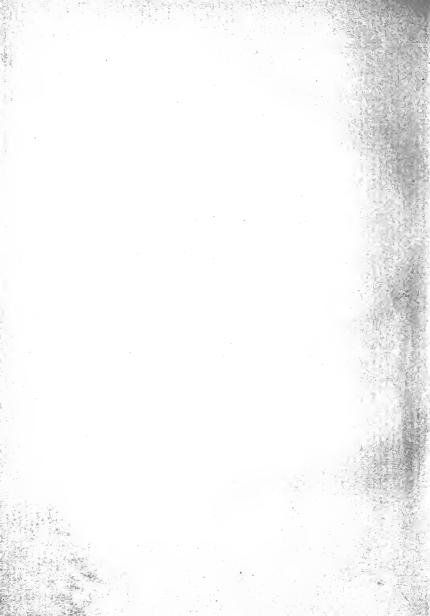
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# LABORATORY NOTES

IN

# HOUSEHOLD CHEMISTRY

FOR THE USE OF

## STUDENTS IN DOMESTIC SCIENCE

BY

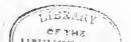
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#### SECOND EDITION

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### Preface to the Second Edition

The favorable reception of the first edition has encouraged the authors to prepare and submit the second revised and enlarged edition; with the hope that it will fully meet all requirements and expectations.

It has seemed best to include in the present edition a large amount of descriptive matter, which was not made a feature of the former edition, as well as specific instructions in the preparation of reagents and a list of the necessary apparatus.

For the guidance of teachers the following list of reference books is suggested:

Elementary Chemistry....Alexander Smith Organic Chemistry.....Perkin & Kipping Industrial Chemistry.....Thorpe

In conclusion, the authors will be glad to receive suggestions for future editions, from any instructors who may use the book.

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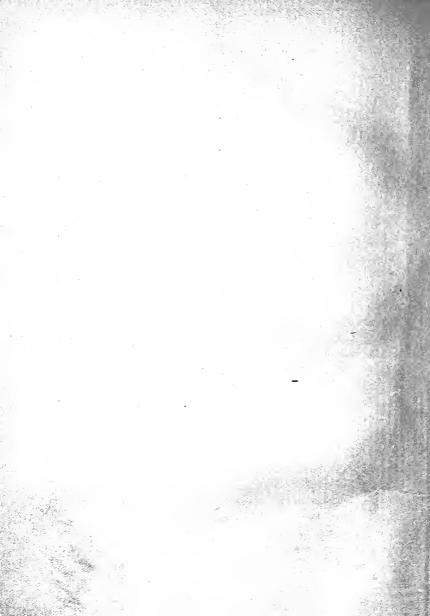
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### Introduction

### CONSTRUCTION OF THE BUNSEN BURNER

Unscrew the tube, examine and light the inner jet. Examine the outer tube and collar that controls the airports. Turn off the gas and replace the tube. Now turn on the gas again, strike a match and approach it to the top of the tube. Always observe this latter precaution when lighting the Bunsen burner. Observe the character and color of the flame, move the collar on the tube and note the effect. Hold a piece of glass tubing near the top of the flame, remove from the flame and bend. Hold it in the same position in the yellow flame, and after removal observe the condition of the tube and try to bend it. Is there any apparent difference in the intensity of the heat developed? Lower a piece of fine iron wire gauze half way in the flame, why does the flame fail to penetrate the gauze? Apply a light above the gauze, explain the phenomenon. Place a piece of paper on the gauze, lower it half way in the flame, notice the charred ring. Hold a splinter at the same point in the flame, note where it is charred and explain. Introduce the large end of a dropping tube

into the flame near the tube, and approach a light near the exit. From the results of the last three experiments what is your idea of the combustion zone?

Carefully turn the gas down at the key, watch the effect, why does the flame disappear? Now immediately turn the gas on full force and note the result. Approach a light to the upper end of the tube, observe the character of the flame, compare with the original flame as to color and heating effect. Strike the rubber tube a quick blow with the closed hand and explain the resulting phenomenon.

Make a simple drawing illustrating the structure of the Bunsen burner, with the gas and air supply and the zones of combustion of the flame.

# INSTRUCTIONS FOR MANIPULATING GLASS TUBING AND CONSTRUCTING SIMPLE APPARATUS

Two kinds of glass, "hard" and "soft," are used in making apparatus for the laboratory. Hard glass is very brittle and quite infusible in the ordinary Bunsen flame. It is used in heavy apparatus where a high temperature is required for heating dry, but never liquid substances, as the latter would cause it to break. It can usually be recognized by the striations on its surface and by its greenish-yellow color, best seen at the end of a broken tube.

Soft glass is less brittle than hard. It is easily fusible

in the Bunsen flame and is used in the construction of thin apparatus (such as beakers, test-tubes, etc.,) for heating liquid, but never dry substances.

The tubing used by the student for bending, blowing and fitting up apparatus should be of soft glass.

Cutting the Tube.—Glass tubing up to one-fourth inch in diameter may readily be cut by making a slight scratch with a triangular file at the point of the fracture, the tube is now grasped firmly in both hands holding the scratch outward and the thumb nails pressed against the inner side of the tube opposite the mark, give a slight bend outward, at the same time pulling apart; the tube will make a clean break and no injury will be received. Broken in this way the tube ends are sharp and should always be rounded by heating for a moment in the flame.

Bending the Tube.—Take care that the tube is perfectly clean and dry inside and outside before heating. Adjust the wing-top to the burner and after lighting, heat the tube lengthwise in the upper part of the flame. Revolve the tube so as to heat all parts equally; when soft, remove from the flame and quietly bend to the desired angle. In case no wing-top is available, the tube may be heated in the same way in an ordinary illuminating burner. The carbon deposited on the tube is readily removed, after cooling, by rubbing with filter-paper.

Drawing the Tube.—Heat as before in wing-top or illu-

minating burner; when soft, remove from the flame and quietly but steadily draw apart. On cooling, the tube may be cut with a file at any spot, and will furnish two pointed tubes. These are used for dropping tubes, by cutting to the desired length and rounding the ends in the flame.

Closing the Tube and Blowing Small Bulbs.—Select a tube with thick walls, cut off a piece about a foot long, heat the square-cut end in the upper part of the ordinary Bunsen flame, revolving the tube continuously while heating; in a short time the tube will close. To blow a bulb continue the heat for a few minutes longer, then remove and blow quietly but strongly into the open end of the tube, continue the air pressure until the desired diameter has been reached, but on no account attempt to make a bulb of more than double the diameter of the original tube, as in this case the walls will be too thin. If it has been impossible to blow a bulb of the desired size in one operation, the tube may be reheated and blown again until the desired diameter has been reached.

Glass rod may be cut, bent and rounded in manner similar to tubing.

#### CONSTRUCTION AND USE OF THE WASH-BOTTLE

Select a clean eight-ounce wide-mouthed bottle, fit to it a rubber stopper pierced with two holes. Now cut two pieces of one-fourth-inch glass tubing, six and ten inches long, heat the longer piece in the wing-top flame about three inches from the end, when soft remove from the burner and bend to an angle of 45°. Heat and bend the shorter piece in the middle to an angle of 135°, round both ends of each tube in the flame, when cold, moisten one end of the short tube with saliva and push it through one hole of the stopper, proceed in the same way with the longer tube, but push it nearly up to the bend, so that when the stopper is inserted in the bottle the other end will just clear the bottom. Cut a piece of black rubber tubing two inches long, slip one end over the longer tube, make a jet by cutting off two inches of the pointed end of a dropping tube, round the rough end, and when cool push it into the rubber tube. The bottle is complete and ready for filling with cold water. By blowing into the short tube, a fine jet of water will issue from the nozzle; by tipping the bottle upside down, a larger stream will issue from the shorter tube.

Wash-bottles for hot liquids are made in the same way, using a thin glass flask instead of a bottle.

# Part I

# Chapter I

#### **FUELS**

Fuels are materials used for producing heat; they must be capable of uniting with oxygen under easily obtainable conditions and of evolving much heat energy during the process of combustion. Occurring as gases, liquids and solids, carbon and its compounds largely fill the required conditions.

Classification: A logical arrangement of the fuels would result as follows:

	Gases	Natural	{ Hydrogen { Hydrocarbons
Pure fuels		Artificial	Carbon monoxide Hydrocarbons
	Liquids	Natural	Hydrocarbons { Alcohols     Hydrocarbons     Anthracite { Coke     Charcoal
		Artificial	
	Solids	\ Natural	
		Artificial	
Impure fuels	Solids	Natural	Soft Coal Peat Woods

Coals, petroleum and natural gas are evidently of plant and animal origin, produced by the natural method of

decomposition similar to the process of dry distillation described on page 9.

The terms pure and impure are used in a restricted sense, the former signifying that the substance is ready for direct combustion, while in the latter case a number of complicated chemical changes must take place before combustion is possible. This is explained in detail in the discussion of the composition of wood.

Historical: Woods both hard and soft and charcoal have been used from the earliest times. Peat, a form of partly carbonized turf, was the main fuel of European countries during the Middle Ages and is still in use. Soft coal came into use during the 15th century, while gas and hard coal were first employed in the early part of the 19th century, and hydrocarbons about the middle of the same epoch. Alcohol is just coming into general use in our own times.

Impure solid fuels on account of more extended use will be first discussed.

### COMPOSITION OF WOOD, PEAT AND SOFT COAL

Wood, peat and soft coal are such impure forms of fuel and must undergo so many and such complicated chemical changes before they are capable of yielding heat, that their actual fuel value is frequently over-estimated and rarely understood by the consumer. The following is a brief and simple statement of composition and changes to be expected.

Wood contains, moisture,  $(H_2O)$ ; resin,  $(C_xH_x)$ ; starch, gum, and cellulose,  $n(C_6H_{10}O_5)$ ; oil,  $(C_xH_xO_x)$ ; mineral matter, or ash.

Considerable heat is required to drive off the moisture and raise the starch, cellulose, etc., to such temperatures that they will decompose, yielding gases of a combustible nature; for example CO, CH<sub>4</sub>, C<sub>2</sub>H<sub>4</sub>, C<sub>2</sub>H<sub>2</sub>, H<sub>2</sub>; in this decomposition H2O is formed and must be driven off as a gas, much heat is also absorbed by the ash in forming new chemical compounds. In fact the fuel efficiency of wood depends entirely upon the relative volumes of combustible gas and charcoal furnished, and as the charcoal or carbon is the best solid fuel, the wood furnishing the largest proportion of carbon in this form is the best fuel, hence we find it advantageous to use hardwood. It must be understood that carbon or charcoal at a red heat combines with a limited amount of oxygen and forms a combustible gas, carbon monoxide, CO, a fuel of the highest heating efficiency.

Soft coal, a partly carbonized plant product, produces less water by chemical change and yields the combustible gases and carbon (coke) in larger proportion.

Hard coal is superior to soft, since it is a purer form of carbon and yields very little combustible gas. It is difficult to maintain the requisite temperature of decomposition during such varied changes, hence we note the dense smoke (carbon) given off by wood and soft coal

fires, this may be partially overcome by adding the fresh fuel in small portions, so that the necessary high temperature may not be lowered. An illustration of the behavior of wood, peat and soft coal is given in the following experiments. The process involved is known as Dry or Destructive Distillation, one of the earliest methods of Organic Analysis, still of great commercial value.

Take a piece of 1/4-inch glass tubing, about I foot long, draw out one end to a fine jet, then heat the tube in the middle and bend at right angles round the large end in the flame and when cold insert it in a perforated cork. Now place 3 or 4 pieces of dry hardwood 1 inch long, 1/4 inch square in a 6" x 1" hard glass test-tube, pushing them down to the end of the tube, insert the cork and clamp the tube and contents in a slightly inverted and inclined position on the ring stand. Gently heat the tube with a broad flame, from the mouth upward. From time to time try the exit tube with moist blue litmus paper, and then with the flame, continue the heating until the pieces of wood appear to be completely charred and no vapor is visible at the exit, but avoid burning the cork. Before allowing the tube to cool, carefully remove the cork, collect any liquid in the tube in another test-tube, close the ignition tube with a fresh cork, and allow it to cool. Observe the odor and general character of the liquid in the test-tube, add an equal volume of water and

shake well, let the tube stand five minutes and filter through with wet paper, test the watery fluid, commercially known as pyroligneous acid, with litmus paper. When the ignition tube is cold remove the charred material, carefully observe its character with a magnifying glass, and make a rough sketch of its structure, float a small piece on water, then boil it for ten minutes, explain the Heat another piece in the flame, held by forceps and finally burn it to ash in a porcelain dish, cool and add water, test liquid with litmus paper, then with a drop of dilute acid, make flame test on resulting liquid with clean platinum wire and blue glass. Make a similar experiment with soft coal; observe that the vapor is first alkaline and finally acid. Note any difference in the products of distillation and ash of soft coal and of wood. Another test should be made with hard coal for comparison. Arrange the results of these experiments in tabular form as follows, noting the comparative quantities of products evolved.

Gas	Vapor	Carbon	Ash
Wood			
Soft Coal			
Hard Coal			

# VALUATION OF COALS AND WOOD FOR FUEL PURPOSES

To determine the value of coal or wood for fuel pur-

FUELS II

poses, proceed as follows: Take one gram of pulverized coal or small pieces of wood in a weighed crucible, dry at 120° C. with cover off, cool and weigh, the loss is water. Heat the crucible with cover on in a strong Bunsen flame for seven minutes, cool and weigh, the loss is volatile combustible matter (tar, smoke, etc.). Heat again with cover off until nothing remains but ash. This operation will require some time; cool and weigh, the loss is fixed carbon (actual fuel). Subtract the weight of the crucible, the difference is ash.

Each student should make a qualitative analysis of the ash of wood, hard and soft coal, from specimens furnished by the instructor, preparing the material according to the following method.—Extract about one gram of the weighed ash on a small (7 cm.) filter several times with 50 cc. of boiling distilled water; mark this water extract and reserve for test. Now extract the residue with the same amount of hot dilute HCl until the final residue is white or nearly so; this is silica or insoluble silicate.

The aqueous and acid solution should be tested separately for the following metal and acid ions: potassium, sodium, calcium, magnesium, iron, sulphates, carbonates, chlorides and phosphates, according to the scheme given below.

SCHEME FOR THE SEPARATION AND DETECTION OF THE ANIONS, CO<sub>3</sub>, PO<sub>4</sub>, SO<sub>4</sub> AND CL AND THE CATIONS FE, CA, MG, NH<sub>4</sub>, NA AND K.

The substance occurring in solid form. In the case of a liquid only the acid need be added.

Treat a small portion of the powder with H<sub>2</sub>O and enough HNO<sub>3</sub> to make the solution acid, boiling toward the close of the operation, only a small residue should remain at this point; if otherwise add a little more acid and boil again, cool and filter, rejecting any residue. The filtrate must be perfectly clear before proceeding with the analysis; if it is not, filter once more. Note whether there is any effervescence when the mixture of water and acid is poured upon the powder; if so, it indicates the presence of carbonates, CO<sub>3</sub>. The gas evolved should be passed into clear lime water, which will cloud if CO<sub>2</sub> is present.

The clear solution is now divided into three parts  $A = \frac{1}{2}$ ,  $B = \frac{1}{4}$ ,  $C = \frac{1}{4}$ .

Operation with solution A 1/2.

Add an equal bulk of ammonium chloride and then ammonium hydroxide,  $NH_4OH$ , until distinctly alkaline (odor of ammonia is sufficient); boil the mixture. If any precipitate forms, filter the mixture; wash with one change of water.

Ppt. ferric hydroxide, a brown gelatinous mass, dissolve this on the filter by pouring over it a small quantity of hot dilute hydrochloric acid; collect the clear yellow filtrate and add to it a few drops of ammonium sulphocyanide; a deep blood red color indicates *Iron*.

Filtrate, clear and colorless, while still warm add ammonium carbonate, shake well, allow the ppt. to settle and cautiously add a little more ammonium carbonate, if no further cloud occurs, enough has been used; now pour the mixture upon a filter and wash with one change of water.

Ppt. calcium carbonate, a white granular mass, dissolve this in the least possible quantity of acetic acid on the filter. To the clear colorless solution add ammonium hydroxide until alkaline, and an equal bulk of ammonium oxalate; boil the mixture. A white granular ppt. of calcium oxalate indicates calcium. This should give a red flame on heated platinum wire.

Filtrate, clear and colorless. Divide into two equal parts A and B.

To A add sodium phosphate and ammonium hydroxide and shake well; if ppt. does not appear at once, cool the mixture, A white crystalline ppt. indicates magnesium. If magnesia has been found in A, pour B into a clean porcelain dish and evaporate off the liquid. Then dry, heat until white fumes (ammonium salts) are no longer evolved. Cool and add a small amount of water, filter, reject the residue. Add two drops of hydrochloric acid to the clear filtrate. Dip in it a clean platinum wire and test in the flame. A vellow color indicates sodium. a violet flame potassium (both to be viewed through blue glass). Potassium may be present even though the flame is yellow; in this case add to the filtrate a few drops of platinic chloride and shake the mixture. A yellow crystalline ppt. indicates potassium.

#### Operation with B 1/4.

Make strongly alkaline with potassium or sodium hydroxide, boil and hold a piece of moistened pink litmus paper in the vapor arising from the boiling mass, being careful that none of it is spattered on the paper. The paper turning blue when moist and back again to pink when dry, indicates ammonia. Where the quantity is large the odor is distinctive.

Operation with C 1/4.

Divide into three equal portions.

Part I.

Add to this a few drops of silver nitrate; a white curdy ppt. of silver chloride, soluble in ammonium hydroxide, indicates Chlorides.

Part II.

Add two drops of hydrochloric acid and a little barium chloride, a white crystalline ppt. of barium sulphate giving a green flame on heated platinum wire indicates Sulphates.

Part III.

Add a few drops (not more than 10) to one inch of ammonium molybdate in a 6 in. tube. Heat the mixture in boiling water about two minutes. A yellow crystalline ppt. of ammonium phosphomolybdate indicates phosphates.

From the results of the above tests the student is required to report any difference in composition of the ash and quantity of ingredients.

The quantity of ash in coals is always greater than in wood, owing to the presence of foreign mineral substances such as silica, lime and sulphide of iron derived from the earthy strata in which the coal is deposited.

Flue dust collecting in stove pipes and flues where hard coal is burned, contains sulphate of ammonia, when

cool this salt absorbs water and attacks iron rapidly corroding the pipes. This fact explains the necessity of cleaning the smoke pipes of furnaces and stoves in the spring of the year when the heating apparatus is no longer used.

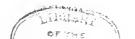
**Experiment.**—Collect some of the light gray dust from a smoke pipe, treat about one gram with boiling water on a filter, pouring the liquid through several times. Reserve the residue and test the liquid in the usual manner for ammonia and sulphates.

Extract the residue still on the filter paper with boiling dilute HCl until the residue is light in color, this is mainly silica from the coal ash, test the acid filtrate for ferric iron and lime in the usual manner.

**Liquid Fuels.**—Hydrocarbons in the form of gasoline or naphtha, and kerosene, and alcohols.

These are highly inflammable liquids distilled from coal or crude petroleum by a destructive process known as "cracking" in which complicated hydrocarbons break up and yield simpler products, any color or objectionable odor is removed by treatment with oil of vitriol (Conc. Commercial Sulphuric Acid) followed by caustic soda and filtration through Fuller's earth.

Benzine an intermediate product while not used as a fuel, serves as a convenient solvent for fats and oils,



and by many is preferred to gasoline for this purpose on account of greater security in handling.

At temperature slightly above normal these hydrocarbons readily combine with oxygen producing intense heat and yielding water and carbon dioxide as products but no ash, with too small supply of oxygen the temperature of combustion is much lowered and a large part of the carbon is not consumed and escapes in a free state producing a yellow flame and if in great excess much black smoke. A very familiar phenomenon in kerosene lamps.

With great excess of oxygen, as when the hot vapor of these liquids is mixed with many times its volume of air, the combustion is so rapid as to produce an explosion (automobile engine). When using these products for fuel purposes care must be taken that these last conditions do not exist. Hence as a measure of safety the lamp or stove reservoir is kept well filled and cool. The following simple experiment will serve to impress these important facts on the student's mind.

Pour not more than one or two drops of clean gasoline into a clean dry wide mouth bottle of 12 to 16 ozs. capacity, stir the vapor for a moment with a hot glass or iron rod and bring a lighted match over the mouth of the bottle, a slight but perceptible explosion should result with or without blue flame.

Pour a teaspoonful of the same liquid in a shallow porcelain dish or saucer, apply the lighted match and

note the yellow flame, but no explosion. Quench by covering with cloth, stiff cardboard or any article that will exclude air.

Gasoline is used quite largely in some localities as a source of heat, being consumed in the so-called blue flame stove which operates by heating the liquid to such a temperature, air being excluded, that vapor forms rapidly and under slight pressure, it is then conducted to the burner (Bunsen) mixed with the proper amount of air and burns with a blue flame.

These stoves and heaters are perfectly safe as long as they are kept clean, do not leak liquid, are kept well filled and furnished with good gasoline.

The quality of gasoline may be determined by the following tests:

- 1. Observe the color, it should be white as water.
- 2. Clearness, if cloudy dirt or water is present; evaporate a small quantity in a clean porcelain dish over warm water (no flame) and examine the residue, also filter some through clean dry chamois skin, water and dirt will remain on the skin. It is a wise precaution for users of gasoline for any purpose to filter as above before using.
  - 3. Test with delicate litmus paper, it should be neutral.
- 4. Determine the specific gravity with the Béaume hydrometer for light liquids; it should register 70-74°.
  - 5. In burning it should give off no odor of hydrogen

sulphide or sulphur dioxide, nor blacken a strip of lead acetate paper held well above the flame.

Kerosene, erroneously called an oil, is much more extensively used and widely known; it is probably the cheapest and best liquid illuminating agent of the present day. The ordinary kerosene wick lamp is so well known as to need no explanation. Kerosene, however, is used in blue flame stoves, such as the Khotal, etc., and although more troublesome to manipulate is preferred by most people because the danger is minimized.

Kerosene should successfully stand tests 1, 2, 3, 5, given under gasoline. The specific gravity should be 48° Beaumé. In addition the following known as the Flash Test is prescribed by the states of New York, Massachusetts, etc.

Flash Test.—Half fill a 200 cc. beaker with kerosene, place over warm water, stir gently with an accurate Fahrenheit thermometer and heat slowly, not more than two degrees rise per minute, until a small open flame brought over the mouth of the beaker causes a blue flame and slight explosion. Note the temperature, it is the flash point and should not be lower than 125° Fahr.

Any oil which will conform to these tests is safe.

Kerosene and gasoline are unsaponifiable: to prove this fact use kerosene, heating a small sample with one-seventh of its volume of strong caustic soda solution

(38°-40° Bé) for ten or fifteen minutes over hot water and stirring often, then allow the mixture to cool, what happens, does the product resemble soap in any way?

Kerosene is often called an oil, is this statement correct? Is it a chemical compound?

Alcohols.—Of this series, only methyl or wood alcohol and ethyl or grain alcohol, are used as fuels. Lately a mixture of the two (90 pts. ethyl, 9 pts. methyl + 1 pt. benzene) has come into general use under the name of denatured alcohol; it is essentially ethyl alcohol and will he treated as such. Methyl alcohol, CH<sub>3</sub>OH or HCH<sub>2</sub>OH, is produced commercially by the dry distillation of wood and known as pyroligneous or wood spirit; it contains light wood tar, acetone, and acetic acid which should be completely removed before using, leaving a bland mild smelling liquid similar to ethyl alcohol known as Columbian Spirit. Much of the ordinary wood alcohol is quite impure. Tar and acetone are easily distinguished by the color and odor, especially if gently heated; acid is readily shown by litmus paper. The supposed poisonous character of methyl alcohol is due entirely to impurities.

As a burning fluid methyl alcohol is distinctly inferior to grain alcohol. The following equation shows the chemical change during complete oxidation:  $2CH_3OH + 3O_2 = 4H_2O + 2CO_2$ .

By partial oxidation, using hot copper oxide, methyl

alcohol is converted into formaldehyde according to the following equation:

CH<sub>3</sub>OH+CuO=CHOH+H<sub>2</sub>O+Cu.

This serves as a valuable test for identifying methyl alcohol and may be carried out in the following manner. Heat a strip of sheet copper red hot in the upper part of the Bunsen flame, and immediately drop it into a test tube half full of the alcohol, wait until the first violent effervescence has passed and then observe the odor of the liquid in the tube and the bright appearance of the copper strip.

In order to obtain formaldehyde in quantity, the warm vapor of methyl alcohol is passed over heated copper oxide in a tube and the product cooled with ice.

Formaldehyde will be treated more in detail under antiseptics.

Methyl alcohol is more volatile than ethyl alcohol, hence the loss in handling and while standing in lamps is greater. By further oxidation methyl alcohol yields formic acid:  $CH_3OH+O_2=HCO_2H+H_2O$ .

Ethyl or grain alcohol: C<sub>2</sub>H<sub>3</sub>OH or CH<sub>3</sub>CH<sub>2</sub>OH. Prepared by the fermentation of glucose or maltose by means of yeasts and distillation of the product. It is a colorless liquid with pleasant and characteristic odor, usually containing about 95 per cent. of pure alcohol and the balance water and small amounts of impurities, acetic acid and acetone more especially; these are not particu-

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larly objectionable if the liquid is to be used for generating heat or general solvent purposes, but in many chemical operations further purification is necessary. Alcohol boiled at 78°-80° C.

When burned in lamps or stoves best of the wickiess type, the following changes take place:

$$C_2H_5OH + 3O_2 = 2CO_2 + 3H_2O.$$

Comparing this equation with that of methyl alcohol on page 19 it will be seen that the amount of CO<sub>2</sub> is doubled, hence it is fair to assume that the heating effect is greater; ethyl alcohol is less volatile than methyl, therefore loss by evaporation during use is less.

Tests on alcohol.

Determine the boiling point of 95 per cent. alcohol by distilling 100 cc. in a small flask fitted with a thermometer and condenser. Determine the acidity of 10 cc. of alcohol with N/10 alkali; report percentage of acidity in terms of acetic acid.

Determine the specific gravity of alcohol by means of the hydrometer and check the result by the Westphal balance.

Heat 10 cc. of alcohol with an equal volume of strong caustic soda (20 per cent.) for some minutes over boiling water, a dark yellow or red coloration indicates aldehyde.

To 10 cc. of alcohol add 1 cc. of iodine solution, heat

gently and add strong sodium carbonate solution slowly until the color disappears, notice the odor of iodoform and when cold examine some of the precipitate under the microscope; pale yellow hexagonal plates appear on the field.

Heat 10 cc. of alcohol with 1 cc. of  $K_2Cr_2O_7$  solution acidified with sulphuric acid, notice the reduction of the chromium to base and the odor of aldehyde.

Dilute I cc. of alcohol to 20 with ordinary tap water, add a small quantity of vinegar ferment, pour the mixture in a small wide-mouthed bottle covering loosely with cotton, and allow the whole to stand several days in a warm place, finally filter and test for acetic acid.

Pure alcohol, free from aldehyde and acid for chemical purposes, can easily be made from the ordinary 95 per cent. variety or even waste alcohol by allowing it to remain for several days in contact with slightly rancid tallow or grease and subsequently filtering, distilling, and neutralizing the product.

On account of the high price, due to the government tax, ethyl alcohol was formerly little used for heat and power purposes, but since the introduction of denatured alcohol, the cost has fallen to fifty cents per gallon and the use enormously increased. At the present price, it is no more expensive to use than gasoline and far safer and pleasanter to handle. Gas consisting of hydrogen, carbon monoxide, and various hydrocarbons

is the ideal fuel. There are five varieties in use, viz.:

Gases proper

{ Natural gas
 Water gas
 Coal gas
 Acetylene
 Naphtha or air gas
 Cold air charged with
 naphtha vapor.

Natural gas is found in large pockets in the earth in many localities; it is reached by drilling in the same manner as for oil or brine. Being under excessive pressure much is lost before it can be controlled and the pressure reduced sufficiently for household use. Consisting mainly of hydrogen and marsh gas, it has little or no illuminating power, but is an excellent source of heat. The supply is gradually being exhausted and many wells have ceased to yield any product. This form of gas has been known from the remotest antiquity, many old shrines being supplied through crevices in the earth, probably the Temple of Diana at Ephesus had a natural gas well.

Fredonia, New York, was lighted with natural gas as early as 1825, the supply being accidentally discovered when boring for salt.

Water or Fuel Gas.—By passing steam at high pressure over white hot carbon Tessie du Motay produced a mixture of hydrogen and carbon monoxide, known as water gas. The equations for the chemical change are as follows:

$$_{4}H_{2}O+C_{2}=_{4}H_{2}+_{2}CO_{2}.$$
  
 $_{2}CO_{2}+C_{2}=_{4}CO.$ 

In order to give the mixture illuminating quality it was passed over gasoline vapor and subsequently through a hot retort to prevent condensation on cooling; the resulting gas was purified in the same manner as coal gas and yielded a product of similar composition containing hydrogen, carbon monoxide, marsh gas, ethylene and acetylene, but in somewhat different proportions and since it contains more carbon monoxide it is generally regarded as a better fuel.

Coal Gas.—From soft or bituminous coals, a gas can be produced by dry distillation, see experiment on page 9. This was the first method used for making gas, and dates back to early days of the 19th century. On account of the many and valuable by-products produced, viz., ammonia, coal tar, carbolic acid, naphthalene, cyanides, etc., it will probably be used for many years to come.

The process consists in heating the coal in large clay retorts, drawing off and cooling the gas in order to condense tar, washing to remove ammonia tar, etc., removing sulphur with lime or iron oxide, storing and delivering the gas under slight pressure. Essentially the same process of purification is used with water gas.

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Acetylene gas, C<sub>2</sub>H<sub>2</sub>, made by the action of water on calcium carbide as follows:

$$CaC_2+2H_2O=C_2H_2+Ca(OH)_2$$
.

Calcium carbide is prepared by heating a mixture of lime and charcoal in the electric furnace.

Either the water is sprayed on the carbide, or finely pulverized carbide is sprinkled in water.

Acetylene is only used in places where ordinary gas cannot be obtained, and is generally used at once. It may, however, be stored in an ingenious manner; strong copper cylinders are partly filled with acetone, and acetylene pumped in until a certain pressure is obtained. By attaching one of these tanks to a lamp, a strong light may be maintained for many hours. The rationale of the process is that acetone dissolves acetylene under pressure and slowly gives it up when the tension is released.

Naphtha Gas.—Many isolated country houses depend for heat and light on this mixture. Outside of the building and underground, is placed an iron tank for holding the hydrocarbon, pipes lead to and from the house, in the house cellar is placed a large revolving drum driven by weights, for forcing air through the gasoline and driving back to the house the vapor laden air. The process is satisfactory on a small scale but rather expensive, depending wholly on the price of the hydrocarbon.

The constituents of illuminating gas are conveniently classified as follows:

Impurities or diluents—oxygen, carbon dioxide, nitrogen.

Illuminants—ethylene, acetylene.

Gas proper—hydrogen, marsh gas or methane, carbon monoxide.

The method of procedure in the analysis is to dissolve out or absorb carbon dioxide, illuminants, oxygen and carbon monoxide in the order named, using the following reagents: caustic potash 20 per cent. bromine, alkaline pyrogallol and cuprous chloride. The insoluble residue consisting of hydrogen, marsh gas and nitrogen, is mixed with oxygen and exploded by the electric spark, which produces carbon dioxide, water and nitrogen. The following equations express the chemical change:

$$2H_2+O_2=2H_2O$$
.  
 $CH_4+2O_2=CO_2+2H_2O$ .

The volume of residual gas is carefully measured and subtracted from the volume before explosion, leaving the contraction, due to H<sub>2</sub>O and designated as C in the following formula. Finally the CO<sub>2</sub> formed is absorbed by KOH, as before, and the loss in volume noted; this known as D in the formula is equal in volume to the original CH<sub>4</sub>.

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The volume of hydrogen is now calculated from these data, according to the formula

$$H = \frac{2(C-D)}{3}.$$

Nitrogen is the difference between 100 and the sum of the other constituents; it should approximate four times the oxygen found.

The value of gas is expressed as candle-power, a standard sperm candle burning two grains per minute being the unit; hence a 25 candle-power gas would give as much light as 25 of the candles burning simultaneously.

The standard illuminating burner consumes five cubic feet per hour under a pressure of one and a half inches of water, this is used as a unit in all gas calculations.

Two styles of meters are used—the wet and the dry; in the former the gas passes through a revolving drum partially submerged in water. The revolutions are registered on dials by appropriate clock work. Since this form of meter is liable to freeze and must always contain water, some of which is lost by evaporation, it has been largely superseded by the dry meter which contains two bellows alternately full and empty. A clock work device, similar to that used in the wet meter, keeps record on appropriate dials. Gas meters are subject to public test and are allowed an error of two per cent. either fast or slow. Hydrogen sulphide is the only impurity in

gas of any importance; by its combustion sulphur dioxide and water are produced, finally resulting in sulphurous acid, which readily attacks fabrics and metals and bleaches many colors. Any hydrogen sulphide escaping combustion, blackens lead acetate paper held above the flame, enough to be influenced by the heat.

Analyses of gas are given below:

Water Gas Per cent.	Coal Gas
Carbon dioxideo.o	0.0
Illuminants 12.6	6.5
Oxygen	0.9
Carbon monoxide 27.3	6.8
Hydrogen 27.7	41.1
Marsh gas27.7	41.6
Nitrogen 3.8	3.7
100.0	100.0
Candle-power 25.04	21.32

In a water gas, the candle-power is usually double the illuminants.

Chemical Changes During Combustion.—The common burner can only use gas of the following composition: methane, CH<sub>4</sub>, ethylene, C<sub>2</sub>H<sub>4</sub>, acetylene, C<sub>2</sub>H<sub>2</sub>, hydrogen, H<sub>2</sub>, and carbon monoxide, CO. Combustion proceeds according to the following equations:

$$CH_4+2O_2=CO_2+2H_2O$$
—heat, no light.  $2H_2+O_2=2H_2O$ —heat, no light.  $2CO+O_2=2CO_2$ —heat, no light.

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 $C_2H_4+O_2=2H_2O+C_2$ —less heat, some light.  $2C_2H_2+O_2=2H_2O+2C_2$ —less heat, more light.

The Bunsen burner mixes the gas with O<sub>2</sub> before combustion; this affects only the ethylene and acetylene as follows:

$$C_2H_4+3O_2=2H_2O+2CO_2$$
—heat, no light.  $2C_2H_2+5O_2=2H_2O+4CO_2$ —heat, no light.

The following simple but important experiments should be performed by each student.

Conditions of Combustion.—Fill a 250 cc. wide-mouthed bottle with four-fifths air and one-fifth gas, collecting it over water, cover with a glass plate and shake thoroughly. Quickly replace the glass plate with wet filter paper, pierce with a pencil point, apply the flame and note the result. Try the same experiment using gas alone. Write equations explaining the results of these experiments.

**Products of Combustion.**—Hold a clean dry bottle for a few moments over a low Bunsen flame and note the result. What compound is formed? Explain and write equation.

Substitute a pointed glass tube for the Bunsen burner, turn the gas low and light at the point, introduce the flame into a clean dry bottle and hold it there for a few moments, note the result and remove the tube. Again introduce it under the same conditions and note the

result. Remove the tube, cover the bottle with a glass plate and turn off the gas. Pour about 10 cc. of lime water, Ca(OH)<sub>2</sub>, into the bottle, shake well, note the result, explain, and write the equation.

Reading the Meter and Testing Burners and Stoves.—Study the dials and figure out how they are read. Connect meter with gas tap and lead rubber tube to burner, be sure the meter is running in the right direction, light the burner and after an interval of a few minutes note the time and meter record, exactly six minutes afterwards again read the meter, subtract the original reading and multiply by ten, the result will be the number of feet per hour consumed.

Heat Consumed in Boiling Water Under Varying Conditions.—Start the burner as in the first experiment and note the time and consumption of gas necessary to boil one quart of water in open and closed vessels made of sheet tin, cast iron, aluminium and copper. Does the shape of the vessel affect the result?

All of the above experiments should be made in a quiet room free from drafts caused by open doors and windows. If stoves are being used they should stand on heavy sheet asbestos in order to avoid loss of heat due to radiation.

# Chapter II

### CARBON DIOXIDE

Carbon dioxide, CO2, erroneously known as carbonic acid, is the chief product of the combustion of carbon and its compounds. It is one of the heaviest of the gases, density 22, or about one and a half times as heavy as air. Under a pressure of 59 atmospheres it liquefies at 20° C., its critical temperature is 31.1° C., sp. gr. of the liquid at 0° C. is 0.95. CO2 dissolves in its own volume of water under N. T. P. conditions. Under a pressure of two or three atmospheres, a solution is obtained familiarly known as soda water. Carbon dioxide is a very stable compound and shows a tendency to dissociate only at very high temperatures: 2CO<sub>2</sub>=2CO+ O2, in the presence of hot carbon this change takes place at lower temperatures (see water gas). Carbon dioxide attacks all metallic oxides in the presence of moisture producing carbonates, particularly the oxides of the alkalies and alkaline earths, viz.: lime, CaO, potash, K2O, and soda, Na<sub>2</sub>O, which are very unstable for this reason.

Carbon dioxide is most easily prepared by attacking marble, CaCO<sub>3</sub>, with dilute nitric acid, HNO<sub>3</sub>, which forms a soluble by-product Ca(NO<sub>3</sub>)<sub>2</sub> and oxidizes the small amount of organic matter present:

$$CaCO_3+2HNO_3=CO_2+H_2O+Ca(NO_3)_2$$
.

Carbon dioxide is also evolved during the growth and development of bacteria, notably the yeasts acting upon soluble carbohydrates, glucoses, maltose, etc.

 $C_6H_{12}O_6$ =2 $CO_2$ +2 $C_2H_5OH$  and utilized in the leavening of doughs and the preparation of effervescing drinks. A mixture of bicarbonate of soda and acid potassium tartrate, under the name of baking powder, when moistened evolves  $CO_2$  according to the following equation:  $NaHCO_3 + KHC_4H_4O_6 = CO_2 + H_2O + KNaC_4H_4O_6$ . Liquid carbon dioxide when released from pressure absorbs much heat in passing into the gaseous state, this fact is utilized in cold storage operations, the gas is collected and recompressed.

The water solution of carbon dioxide is feebly acid due to the formation of carbonic acid,  $H_2CO_3$ , as follows:  $H_2O+CO_2=H_2CO_3$ .

Carbonic acid acts on all carbonates, forming bicarbonates in most cases more soluble in water than the corresponding carbonates; familiar examples are the bicarbonates of lime and magnesia, causing the hardness in water CaCO<sub>3</sub>+H<sub>2</sub>CO<sub>3</sub>=Ca(HCO<sub>3</sub>)<sub>2</sub>, and baking soda Na<sub>2</sub>CO<sub>3</sub>+H<sub>2</sub>CO<sub>3</sub>=2NaHCO<sub>3</sub>.

At or near the boiling point of water, carbonic acid breaks up into CO<sub>2</sub>+H<sub>2</sub>O, likewise the bicarbonates decompose into carbonates, water and carbon dioxide;

hence all soluble bicarbonates should be dissolved in cool water.

Preparation of Bicarbonate of Lime.—Dilute 50 cc. of clear fresh lime water, Ca(OH)<sub>2</sub>, with an equal volume of water and pass a rapid current of CO<sub>2</sub> through the mixture. What is the white precipitate? Write the equation. Why does the white solid disappear after the gas has been passing for some time? Write the equation. When the liquid is perfectly clear, test with litmus paper, and then boil one-half of it. Explain the result by means of an equation.

This experiment has a special bearing on the softening of water.

Formation of Sodium Bicarbonate.—Dilute 25 cc. of strong ammonia with an equal bulk of water, cool and saturate the liquid with salt, NaCl, then pass a rapid and continuous current of carbon dioxide through the solution and keep up the action until no more precipitate forms, filter and wash with a saturated solution of sodium bicarbonate, dry and weigh the resulting NaHCO<sub>3</sub>. Complete the equation:

NaCl+NH<sub>3</sub>+H<sub>2</sub>O+CO<sub>2</sub>=NaHCO<sub>3</sub>+.

The special functions of carbon dioxide to plant and animal life will be discussed in the next chapter.

# Chapter III

### THE ATMOSPHERE

Composition.—Pure air is a mixture of four parts of nitrogen and one part of oxygen. Other constituents occur in small quantities, chief of which are water and carbon dioxide. The density of the air is approximately 14.5 in the dry state and it will support 30 inches or 760 mm. of mercury at the sea level. By admixture with water vapor density 9, its weight and supporting power decline, hence the barometer does not stand as high during the period just preceding a storm. Evidently the expression heavy is misplaced in this case. The pressure of the atmosphere on each square inch of surface is fifteen pounds, but is rarely appreciated, since it is exerted in all directions. The following simple experiment graphically demonstrates this pressure: Pour two inches of water into a clean ordinary half gallon can, boil vigorously and close the opening with a close fitting cork. Remove the burner and when cool the can will collapse. The can should have a small opening and preferably be rectangular in shape.

Air is slightly soluble in water, but its composition is markedly different from the atmosphere, containing more CO<sub>2</sub> and having its oxygen and nitrogen in the propor-



#### THE ATMOSPHERE

tion of 1-3. Its exhilerating effect in water is well known. Air is liquefied at a temperature of -190° C.

Carbon dioxide occurs in air to the extent of 0.03 per cent. A portion of this is washed by the rain and combines with lime forming the shells of molluscs; the larger part, however, is utilized by the plant organism in the formation of starch and storing of the sun's energy. This subject will be discussed more in detail under carbohydrates. The dust of air is very variable in composition and quantity but may be classified as

Mineral—particles of soil, iron, glass, carbon, etc. Organic—Dead—fragments of plant, hairs, etc. Living—pollen, spores, and bacteria.

The dust in air, irrespective of quality, curiously influences the deposition of excess moisture, causing it to condense in visible particles; hence we have fog, more prevalent the greater the amount of dust in the locality, as in large manufacturing cities.

Dustless air can be prepared by sucking air into a flask through a tube 12-15 inches long filled with cotton.

Examine the dust sweepings from a window sill, look for iron with a magnet, burn off the organic matter and note the mineral residue, extract some of this with boiling dilute HCl, note the white residue of silica and test the solution for iron and lime.

A little of the original dust sprinkled into milk will

cause it to decompose quickly. It will ferment weak sugar solution and cause stronger ones to mould. Try these experiments.

Do not fail to carefully examine a minute portion of moistened dust under the microscope with low power. Each student should make the following tests:

- 1. Pour an inch of alkaline pyrogallol into a short broad test tube, close with a rubber stopper, invert and mark the position of the stopper and liquid on a gum label pasted on the outside of the tube, shake the tube well, invert and open under water, mark the level of the water in the tube when open, and explain the phenomenon.
- 2. Fasten one inch of Christmas candle to a flat cork, float on a shallow dish of lime water, light the candle and invert a clean dry beaker over it, add more lime water if necessary. Describe and explain the result. Try a lighted candle in the residual air, what is the result? Explain.
- 3. Carbon Dioxide, CO<sub>2</sub>.—Expose a few drops of lime water on a slide to the air and notice that, by the end of the lesson, it is cloudy. Examine under the microscope the rhombohedral crystals of calcium carbonate, CaCO<sub>3</sub>, and draw a diagram of them.
- 4. Hydrogen Sulphide,  $H_2S$ .—Moisten a filter paper with a solution of acetate of lead and expose to the air until the end of the lesson. Notice the black coloration due to the formation of lead sulphide. This test works very well in gas-lit rooms.

5. Expose strips of paper, saturated with covalt chloride or iodide and then thoroughly dried, to the air out and in doors; under moist conditions, it turns pink.

Weigh out a small watch glass containing about one gram of fused calcium chloride, wait about two hours and weigh again. Note the increase in weight largely due to water.

Special functions of the air constituents with regard to plant and animal organisms. Oxygen of the air inhaled by the warm blooded animals combines with the haemoglobin in the lungs is carried to the tissues where oxidation takes place and the resulting carbon dioxide brought back to lungs and released. Not all of the oxygen inhaled is absorbed as the exhaled breath of human beings contains in 100 cc. 15.9 cc. of oxygen and 3.7 cc. of carbon dioxide. The carbon of the carbon dioxide is converted by the plant into carbohydrate which it retains and the free oxygen restored to the air.

Water serves as a circulating medium and solvent for both plants and animals, the former specially need it for complicated changes, explained later under hydrolysis.

Water on account of high heat capacity acts as a temperature regulator, the bodily temperature being controlled by evaporation from the skin. Most of the bacteria are harmless and useful—such as yeasts, lactic and acetic acid, etc.

# Chapter IV

### WATER

Physical Properties.—Water, hydrogen monoxide, H<sub>2</sub>O, the universal solvent, exists in three states without change of composition; as a gas, (steam) at 100° C., as a liquid (water) between 0° and 100° C., maximum density, 4° C., and as a solid (ice) below 0° C. In passing into the gas state, the water absorbs 537 calories (a calorie is the amount of heat necessary to raise the temperature of water one degree centigrade), and expands from 1 to 1728 volumes, hence the expression a cubic inch of water will make a cubic foot of steam.

In the liquid state, water is practically incompressible, but passing into the solid state, it expands 10 per cent. yielding up 79 calories.

At all temperatures, water passes into the vapor state depending upon the condition of the surrounding atmosphere, whether saturated or not.

Water is used as a standard of weight, one cubic centimeter at 4° C. being called one gram. In the common system, one pint of water weighs approximately one pound, and one U. S. gallon, 231 cubic inches, containing eight pints. The weight of one gallon is about 8.3 lbs. About 7.5 gallons make a cubic foot.

The boiling and freezing points of water are used as convenient points for standardizing thermometer scales; in the centigrade system 0°-100°, in the Fahrenheit 32°-212°, in the Rèaumur 0°-80°, all under ordinary atmospheric pressure. As a standard unit for specific gravity measurements, water serves for liquids and solids. The well known hydrometer, an instrument for determining the sp. gr. of liquids, floats at mark 1 in distilled water of 15.5° C. or 60° Fahr. On the Béaume scale water is marked as zero.

Pure water is a poor conductor of electricity and heat, but dissolved matter increases its conductive capacity.

Chemical Properties.—As a chemical agent, water is extremely potent, acting usually as a solvent, but in many cases producing profound chemical changes. Briefly the action of water may be classed as follows:

Water of solution, water of hydration, water of hydrolysis.

Water of Solution.—When any solid, salt, niter, etc., dissolves in water, loss or gain of heat is apparent, but on evaporating the liquid, the solid reappears in the original form.

Water of Hydration.—The soluble substance eventually reappears on partial evaporation of the liquid in changed form containing some of the water in the solid state and known as water of crystallization. Familiar examples are washing soda, alum, Glauber's salt, etc.

Water of Hydrolysis.—By double decomposition, the water molecule appears as OH, hydroxyl, such as solution of caustic alkalies and slaking lime:

$$Na_2O+H_2O=2NaOH$$
.  
 $CaO+H_2O=Ca(OH)_2$ .

Or the solution of non-metallic oxides,

$$SO_3$$
,  $N_2O_5$ ,  $P_2O_5$ :

$$SO_3+H_2O=SO_2(HO)_2$$
 or  $H_2SO_4$ .

$$N_2O_{l5}+H_2O=2NO_2HO$$
 or  $2HNO_3$ .

$$P_2O_5+3H_2O=2PO(HO)_3$$
 or  $2H_3PO_4$ .

and the change of starch, sugar, etc.:

$$C_6H_{10}O_5+H_2O=C_6H_{12}O_6$$

$$C_{12}H_{22}O_{11}+H_2O=2C_6H_{12}O_6.$$

Natural Waters, Classification.—Natural waters are never pure, dissolving or holding in suspension gases, liquids and solids with which they come in contact. The following is a convenient classification:

Atmospheric Snow Contain very little dissolved solids but dust and gases of the atmosphere.

Natural Waters.—

Salt

Surface — usually a large amount of suspended matter.

Terrestrial Sweet Underground—clear, minimum of suspended matter.

Brines—over 5% soluble salts.

Sea water—3.6% solids.
Excess of mineral matter and gases.

Mineral

Potable or drinking water should be clear, free from odor and color, and should contain not more than twenty grains of solids per U. S. gallon, of which not more than one-half is organic matter.

The soluble mineral matter in water consists of a mixture of the following salts:

Carbonates )		Sodium
Bicarbonates	of -	Potassium
Sulphates		Calcium
Chlorides		Magnesium

together with oxide of iron and silica in minute amounts. An excess of chlorides may be due to sewage or animal contamination, excess of lime causes hardness, and excess of iron usually is apparent from the color and is probably due to the solvent effect of organic matter in the water.

On boiling, water loses its dissolved gases, hence distilled or sterilized water is flat or stale.

### EXPERIMENTS ON WATER

1. Heat Conductivity.—Fill an eight-inch test tube two-thirds full of water, grasp the lower end of the tube with the fingers and hold in the flame at a slight inclination from the perpendicular. Note that the upper part will boil before the lower becomes uncomfortably hot to hold. Reverse the order of heating and note the same result. Explain.

- 2. Boiling Point Under Atmospheric Pressure.—Pour about 250 cc. of distilled water into a half liter round bottom flask supported on a right stand. Introduce a thermometer so that the bulb only is immersed in the liquid and apply heat. Note the point to which the mercury rises when the liquid is quietly boiling, raise the thermometer bulb just out of the liquid and boil again. Is there any difference? Does the thermometer indicate any higher degree of heat when the liquid boils violently?
- 3. Boiling Point Under Reduced Pressure.—Select a cork which fits the flask tightly, pierce a hole through it and insert a thermometer. Now half fill the the flask with water and boil the liquid. When in active ebullition, close the flask with the cork and thermometer and withdraw the heat. In a few minutes the liquid will cease to boil, then read the thermometer and grasping the neck of the flask with several folds of a towel hold it under the cold water tap, what happens? Read the thermometer and explain.
- 4. Freezing Point.—Place a six-inch funnel in the ring stand with drip cup beneath, suspend a thermometer on the same stand so that the bulb is half way down the funnel. Fill the funnel with finely cracked ice and note the lowest temperature reached by the thermometer. Repeat the experiment, using fine salt with the ice. Why does the mercury go lower?

The foregoing experiments 2, 3 and 4 serve as excellent methods of testing the accuracy of thermometers.

- 5. Influence of Soluble Matter.—Repeat experiment 2, after dissolving two tablespoonfuls of salt in the water. Note the temperature at which the liquid now boils, cool and take its sp. gr. and reserve for use in experiments 6 and 14.
- 6. Take the larger part of the liquid prepared in the previous experiment and boil it down in a small beaker to one-half of its bulk, how does the thermometer stand now, remove it and allow the liquid to cool thoroughly. Examine the crystalline deposit with a lens, draw a diagram of what you observe. Taste the residue. Does it suggest the original salt?
- 7. Make a strong solution of sugar in water, take the sp. gr. of the cold liquid and treat it in the same way as the salt solution in experiment 6.
- 8. Note the boiling point of a mixture of equal volumes of water and strong alcohol. Preserve the liquid and return it to the instructor.
- 9. Water as Solvent.—Determine the weight of salt necessary to make a saturated solution (pickle) at ordinary temperatures. Use cold water and shake well. Take the sp. gr. of the liquid with a Beaumé hydrometer and record the result. Will a fresh egg sink or float in this liquid? Try it.

- spoonful of dry pulverized lime, (CaO), add to it an equal volume of cold water, stir the mixture in a small porcelain dish with a thermometer, adding more water if necessary, record the thermometer readings carefully. At the conclusion of the experiment, wash the material into a wide-mouthed bottle, fill up with distilled water, cork and shake well, let it stand until clear and then carefully pour away the liquid, add more water, cork and shake well again, reserve for future use. The second clear solution is called lime water, Ca(OH)<sub>2</sub>, and is much used in the laboratory and household as a mild alkali, try it with litmus paper, also taste the clear liquid.
- 11. Take a tablespoonful of common plaster, mix this with half the volume of water in a porcelain dish, stirring as before with a thermometer, record the result and compare with experiment 10.
- 12. Slowly pour about 10 cc. of strong sulphuric acid, H<sub>2</sub>SO—into 50 cc. of cold water, stir well with a thermometer and from time to time record the temperature.
- 13. Carefully mix exactly 9 volumes, (45 cc.) of alcohol,  $C_2H_5OH$ , and 1 volume (5 cc.) of water. How many volumes result? Use the burette and cylinder. Record and explain.
- 14. Effects of Filtration.—Add a few drops (10) of the liquid from experiment 5 to a large volume of water,

(50 cc.), filter and taste the liquid. Is any change produced? Now add to the filtrate a few drops of silver nitrate, AgNO<sub>3</sub>, shake well and filter again, note any difference.

- 15. Dissolve a few grams (1-2) of copper sulphate in 250 cc. of water; enough should be used to give the resulting liquid a distinct but not deep shade. Filter a little of this, what is the result? Reserve the bulk for experiment 18. What inference do you draw from experiments 14 and 15.
- 16. Charcoal Filtration.—To 50 cc. of water add enough caramel solution to give it a distinct but not deep yellow color, then divide into two equal parts. Filter one through dry freshly ignited bone-black several times and compare the color of the resulting liquid with the original solution.
- 17. Effect of Alum.—Take any sample of cloudy or slightly colored water, even soapy water will answer. Add a very small quantity of finely powdered alum, shake well, filter and compare with the original sample.

Water should be neutral or slightly alkaline to work well with alum.

18. Distillation of Water.—Place on a wire gauze or sand-bath and make firm with a clamp, a round bottom half liter flask containing about 250 cc. of the liquid made in experiment 15. Insert in the

flask a cork fitted with a thermometer and 45 degree exit tube, and connect this latter with a straight tube air condenser three feet long. Boil the liquid slowly, taking note of the boiling point of the liquid. Carefully examine the distillate, see if you can detect any traces of the CuSO<sub>4</sub>, if so moderate the heat, collect another portion and test it for acidity. Was the original liquid acid? Explain. Remove the burner, cool the apparatus and add 5 cc. of ammonia, shaking well after adding the alkali, a deep blue color should be obtained. Distill this liquid and test the distillate as before.

### WATER ANALYSIS

Qualitative Examination of Water.—No attempt will be made in this work to give methods for quantitative analysis of the impurities found in water. We wish, however, to give certain qualitative tests which will aid in detecting such impurities, when present in abnormal amounts; it is only when found in abnormal amounts that the water is open to suspicion. The impurities are for the most part harmless in themselves. A thorough investigation of the surroundings and of the sources of contamination of the water supply, care in taking the sample, and other precautions are quite essential.

The tests usually made are—color and appearance, odor and taste, and for the presence of total solids, free and

albuminoid ammonia, nitrogen as nitrites and nitrates, chlorine, temporary and permanent hardness, and sometimes phosphates, sulphates, etc.

Total Solids.—Evaporate to dryness in a clean porcelain dish 100 cc. of ordinary drinking water. Examine the residue, if any, and notice if it blackens on heating. This indicates organic matter. Phosphates may be determined at this point by dissolving the residue in a little hot water, and adding the solution to a mixture of nitric acid and ammonium molybdate; if phosphates are present, a yellow color or crystalline precipitate will be formed on gentle heating.

Ammonia.—Two forms of ammonia are looked for in water, the "free ammonia" and the so-called "albuminoid ammonia."

Free Ammonia.—This is determined by distillation as in experiment 15, omitting the copper sulphate, and testing each 20 cc. of the distillate with Nessler's solution; which gives a yellow or brown color in the presence of ammonia. Continue until a portion is found which fails to respond to the test; at this point the water is ammonia free.

Albuminoid Ammonia.—This is ammonia derived from organic matter, by means of alkaline permanganate of potassium. It may be applied to the water already in the flask from which the free ammonia has been ex-

pelled or to a fresh sample, in which case you obtain the total ammonia and deduct the free ammonia.

Test.—In either case, add to the water in the distillation flask 10 or 15 cc. of alkaline permanganate, and test the distillates as before with Nessler's solution. Note the difference in the amount of ammonia set free.

Nitrites.—The presence of nitrites in water is supposed to be due either to the reduction of nitrates already present in the water by the action of organic matter, or to the oxidation of organic nitrogen to nitrite.

Test.—To about 100 cc. of water in a Nessler tube, add 5 cc. of a freshly prepared mixture of equal parts of sulphanilic acid dissolved in acetic acid and naphthylamine acetate dissolved in dilute acetic acid, mix and allow to stand for thirty minutes. If the solution becomes pink the water contains nitrites. Compare with water known to be nitrite free.

**Chlorine.**—Chlorine is found mostly as sodium chloride, although other chlorides may be present.

Test.—Place in a small casserole or porcelain dish about 100 cc. of the water to be tested, and in another dish the same amount of distilled water. Add to each, two or three drops of potassium chromate solution, then add drop by drop a dilute solution of silver nitrate (N/10) stirring after each drop until a faint tinge of red remains. Obtain the same tint in each, and note the

number of drops of silver nitrate used in each case. The difference between the two shows the amount of chloride present. (Each drop of silver nitrate solution is equivalent to 0.000293 gram sodium chloride). Excess of chlorides usually indicates sewage contamination.

Hardness.—By hardness is meant the soap destroying capacity of a water. This property is due principally to the fact that calcium and magnesium salts form, with ordinary soaps, insoluble compounds or soaps which separate as a curd in the water and have no detergent value. The hardness of a water may be classified under two heads, viz., "Temporary" and "Permanent."

Temporary Hardness.—Temporary hardness is caused by the carbonates of calcium and magnesium held in solution as bicarbonates by carbonic acid present in the water. Boiling expels the CO<sub>2</sub> causing a precipitation of calcium and magnesium carbonates.

**Test.**—Place 100 cc. of the water to be tested in an eight ounce bottle, add an alcoholic solution of pure castile soap one-half cc. at a time, shaking thoroughly after each addition, until a lather is formed which lasts five minutes. Note the amount of soap solution used.

Now boil 100 cc. of the sample, cool and repeat the test, noting again the amount of soap solution used. The difference gives the temporary hardness.

Permanent Hardness.—Permanent hardness is due to

the presence of calcium sulphate and other soluble salts of calcium and magnesium, not carbonates, hold in solution by the solvent action of the water itself. Such a water cannot be materially softened by boiling but may be softened by boiling with sodium carbonate, which converts the sulphates, etc., into carbonates and precipitates them as such.

Experiment 1.—Prepare hard water by dissolving 0.100 grams of plaster (calcium sulphate) in 500 cc. of distilled water. Pass a rapid current of carbon dioxide through the solution and continue, at least as long as in the case of lime water (page 33). Why is the action not the same? Finally add sodium carbonate solution and note the result. Explain.

Most waters possess both temporary and permanent hardness, in which case the total hardness is first determined. The water is then boiled and the permanent hardness determined, the temporary hardness being obtained by difference.

**Oxygen Consuming Power.**—Generally ascribed to organic matter.

Fill two clean eight inch test-tubes, one with the water to be tested, the other with distilled water, add to each the same amount of acidified potassium permanganate solution. Be careful not to obtain too deep a shade and see that the shades match. On standing ten minutes, there should be an appreciable lightening in color, greatest in the tube of water under test. If the color entirely disappears, the amount of organic matter is probably dangerously great. Compare with the test under total solids.

# Chapter V

### THE METALS AND ALLOYS

Only those in common use will be considered. The list includes:

Iron, in the form of wrought and cast iron and steel. Zinc. rolled or sheet and cast.

Copper.

Lead, as sheet or pipe.

Aluminium, cast and rolled.

Nickel—Also various compound forms, as follows: Tin plate, thin sheet iron or steel coated with molten tin.

Galvanized iron, cast or wrought iron or steel coated with molten zinc.

The alloys are solid solutions of metals and constitute a valuable member of this group. Generally speaking, their properties are similar to that constituent which forms the larger part of the mass, but they are usually harder and have a lower melting point.

Familiar alloys of silver are "sterling" and "coin;" of copper, brass, bronze, and German silver; of lead, solder; of tin, pewter, and brittania metal. All the metals and alloys are subject to oxidation or rusting in the presence of air, moisture and carbon dioxide, but with the ex-

ception of iron, the action is superficial and not progressive, the coat of oxide protecting the metal beneath from further action.

Iron, (Ferrum) Fe, occurs in nature largely in the form of oxides: haematite, Fe<sub>2</sub>O<sub>3</sub>, (red), and magnetite, Fe<sub>3</sub>O<sub>4</sub>, (black), the latter possessing magnetic qualities and commonly called "Lodestone."

The metal is obtained by fusing the ore in shaft furnaces with excess of carbon and enough limestone to furnish a fusible ash or slag with the silicious matter present in the ore. The following equations explain the reduction and slagging.

$$2Fe_3O_4+8CO=3Fe_2+8CO_2$$
.  
SiO<sub>2</sub>+CaO=CaSiO<sub>3</sub>.

The product "Pig Iron," or crude cast iron, contains from 3-4 per cent. of carbon as graphite and combined carbon or carbide of iron Fe<sub>3</sub>C, rendering the mass fusible. By careful smelting in small shaft furnaces called "Cupolas," the pig iron is obtained in the form of gray, white and mottled iron, depending on the rapidity of cooling the moulds. Pig iron frequently contains small amounts of impurities, sulphur and phosphorus, rendering the product short or brittle, while hot or cold; during the refining process these are almost entirely removed in the slag. Cast iron is brittle and hard, it melts without softening at 1200° C, and yields a thin

liquid which may be cast in sand moulds. The quality of the product depends largely on the purity of the iron, (freedom from S. and P.), its temperature of cooling, and the smoothness of the mould. If cast iron be subsequently and repeatedly heated to redness in presence of air, it becomes much harder and more brittle, frequently cracking under careless handling as in overheated stove lids, etc.

Cast iron heats more slowly but retains its heat better than other forms of the metal, hence its use for oven plates, sadirons, stove lids, etc. It does not oxidize as easily as steel or wrought iron.

Malleable iron is a form of cast iron which has been slowly cooled and thereby has gained a certain degree of elasticity. It is softer and less brittle than ordinary cast iron and is much used in house hardware.

Wrought iron and steel are prepared from pig iron by burning out the carbon in hot air furnaces of special construction.

The reverberatory furnace for producing wrought iron is really a large oven heated by gas and provided with a powerful blast of hot air. Liquid pig iron is run on to the hot furnace bed where the excess of oxygen removes the carbon as follows:  $C_2+O_2=2CO$ . As the CO escapes from the liquid mass it produces a bubbling like any boiling liquid. Gradually as the carbon is burned

out, the iron becomes pasty or semi-solid and is collected in balls with large pokers operated by hand (puddling). When the balls are of sufficient size they are removed with large tongs, squeezed to remove slag and rolled into short bars (blooms or billets). The blooms are then reheated until soft and rolled in bars and rods; when cold the bars may be drawn down through steel dies into wire of almost any degree of fineness. The bars are cold forged into nails and tacks. Piano wire, the purest form of iron, contains 99.7 per cent. Fe, the balance is mainly carbon. Cold wrought iron is quite soft, bends easily and has great tensile strength. It does not melt readily (1600° C.), but softens on heating and may be forged and welded.

Steel is a form of iron between cast and wrought, containing 1.5 per cent. carbon. When heated and slowly cooled it is soft, (mild) but if suddenly cooled is harder than glass. Hardened steel cautiously reheated, may be softened to any desired extent. (Tempering). At a high temperature steel melts and may be cast like iron.

Two kinds of steel are manufactured, *i. e.*, Bessemer, the cheaper variety used for rails, plate for making so-called sheet tin and galvanized iron, when nails, etc., and open hearth steel, a more expensive variety used for cutlery and tools.

Bessemer Process.—The cast iron is first melted in a cupola, and then run into a special furnace (the Con-

verter), where a powerful blast of hot air bubbles through the molten liquid and quickly (15 minutes) burns out the carbon and other impurities and even produces some oxide. Just at this point, a small portion of molten cast iron containing manganese and the proper amount of carbon is added and the mixture immediately poured into the moulds and cooled. The function of the manganese is to assist in holding the carbon in solution.

Open Hearth Method.—The cast iron is melted in a gas furnace with dish-shaped bed together with scrap wrought iron and iron ore. After eight or ten hours heating, the operation is complete and the liquid steel is drawn off and cast in ingots.

Steel rusts much more readily than cast iron and usually needs, especially if polished, a protecting coat of oil. Rust may be removed by soaking in kerosene and rubbing with fine emery or carborundum, and oil.

Rusting of Iron.—Two forms of oxide may be produced, one the common red or brown rust, FeO,  $Fe_2O_3+H_2O$  forms in the air in presence of moisture and  $CO_2$ . It is soft and friable and does not protect the metal from further action. It is slightly soluble in water, giving it a characteristic taste, experienced in drinking water conducted by iron pipes. The other type of oxide,  $Fe_3O_4$ , is formed by the oxidation of hot iron, (Russia Iron), or by the action of superheated steam and carbon monoxide (Barff Process).  $Fe_3O_4$ 

forms a dark gray adherent but brittle coat and protects the metal from further action.

## EXPERIMENTS ON IRONS AND STEEL

**Physical Properties.**—Notice their magnetic qualities using an ordinary horse-shoe magnet and some coarse needles, pieces of soft iron wire and cast iron.

Heat small pieces of the various kinds of iron in the Bunsen flame and note their behavior, (time of heating and cooling).

Temper and draw temper of a knitting needle.

Test the hardness of a file on glass. Break off one end of a small round file and examine the fracture with a strong lens.

Chemical Properties.—Boil small pieces of bright iron (wire or cast) in the following liquids: Dilute mineral acid, organic acid (acetic and tartaric) and caustic soda. Record the results. Filter off the liquid in each test and make it acid with HCl and add NH<sub>4</sub>CNS. A blood red color shows iron in solution. Treat rusty iron in the same way and record results. Expose small pieces of bright iron in two separate bottles, one containing a small amount of water and the other water and carbon dioxide. Cork both and set aside. Which rusts first, why?

Nickel, a hard silver white metal only slightly susceptible to oxidation in moist air, is largely used as a protective and decorative coating on iron and copper. Pure nickel utensils are used in the laboratory taking the place of iron, but comparative high cost has prevented their employment in the house. As an ingredient of alloys, nickel is found in German silver and coin nickel.

The process of plating is as follows: Prepare an ammoniacal bath of ammonium nickel sulphate, suspend in it the article to be plated which must be thoroughly cleaned by dipping in acid, brushing and rinsing in water. This forms the cathode and a nickel plate the anode.

Nickel plated articles should always be cleaned with a mixture of diluted ammonia and whiting, or rouge, and polished with soft cotton waste.

**Experiment.**—Heat small pieces of pure nickel with dilute acids and alkalies as under iron and record the results. See page 57. Soluble salts of nickel have a green color and yield a black precipitate NiS with ammonium sulphide.

Aluminium (incorrectly aluminum) the most abundant of the metals, a constituent of common minerals; Feldspars, micas, clays and cryolite. The metal is prepared by electrolysis of the oxide in a bath of molten cryolite.

Aluminium is silver white in color, almost as hard and tenacious as steel, melts at 600-700° C. and does not tarnish readily. On account of its lightness it is much in demand for cooking utensils but care must be taken

that it does not come into contact with acids and caustic alkalies, which vigorously attack it, liberating hydrogen and forming soluble aluminium compounds.

Experiment 1.—Heat several strips of thin sheet aluminium in a large test tube with 10 per cent. caustic soda solution. Note the violent effervescence; approach a lighted match to the mouth of the tube. What happens? Explain. Before the metal has entirely disappeared, cool the tube, add an equal bulk of water, filter the liquid and exactly neutralize the filtrate with dilute HCl. Note the copious flocculent precipitate,  $Al_2(OH)_6$ . Repeat the experiment using HCl instead of alkali and finally neutralizing with ammonia. Write equations explanatory of the chemical changes observed.

Aluminium articles should never be washed with strong acids or alkalies, rather use neutral soap with a small addition of ammonia and polish with some smooth powder such as whiting or rouge. Powdered aluminium mixed with oxidized linseed oil and amyl acetate or acetone is largely used as a bronze paint.

Two alloys are known; magnalium, containing 6-30 per cent. of magnesium, taking a high polish and working well in the lathe and aluminium bronze, 88-95 per cent. of copper, easily fusible, golden in color and of great strength and resisting qualities.

Zinc occurs chiefly as Calamine or Zinc blende ZnCO<sub>3</sub>. After calcination to drive off CO<sub>2</sub>, the oxide is mixed

with carbon and distilled in earthen retorts at 1300-1400° C.; crude metallic zinc "spelter" condenses in the receivers and CO burns at a small opening.

$$ZnCO_3=ZnO+CO_2$$
.  
 $2ZnO+C_2=2Zn+2CO$ .

Zinc is bluish white, highly crystalline and brittle when cold. By heating to 120-150° C. and rolling under hot rolls it remains pliable and soft on cooling (sheet zinc). At 200-300° C. it becomes brittle again, melts at 433° C. and boils at 920° C.

Zinc burns in the air with a bluish white flame yielding a white oxide (used as a base for print).

In moist air, it oxidizes and absorbs CO<sub>2</sub>, forming a thin adherent coat of basic carbonate which protects the metal from further change. Dilute acids readily dissolve this coating and thus restore the original brilliancy. Acids and alkalies freely attack zinc, liberating hydrogen and producing soluble compounds which are poisonous, hence zinc vessels should never be used for the preparation or storage of food.

Sheet zinc is frequently used for roofs, gutters, cornices and leaders of buildings; but does not last well near the sea shore, on account of the salt in the atmosphere.

The molten metal mixes in all proportions with copper, tin and antimony, see German silver, brass, etc.

Zinc, both cast and rolled, is largely used in primary batteries. It lasts much better if cleaned with dilute sulphuric acid and coated with mercury (amalgamated). Do not attempt to cleanse zinc or galvanized iron with anything but neutral soap and hot water.

Experiment 1.—Immerse strips of sheet zinc in a dilute solution of sodium chloride for several days, finally remove and examine the strips for corrosion and test the liquid with hydrogen sulphide for soluble zinc. The action will be much more apparent if clean iron or copper nails are in contact with the zinc.

**Experiment 2.**—All kinds of iron are galvanized or coated with zinc by first cleansing the article thoroughly with acid, draining and then dipping in molten zinc. Try it.

**Experiment 3.—**Treat small pieces of sheet zinc with dilute acids and alkalies, see experiments on iron, page 57, and record the results.

Copper, Silver and Gold.—Copper, cuprum,¹Cu, is found native, also as sulphide and carbonate. Native copper ore is crushed, washed to remove rock and melted with flux. The metal usually contains a small amount of silver which is removed by electrolysis. Carbonates and oxides are fused with coal to reduce the metal. Sulphide ores re-

<sup>1</sup>The term "Cuprum" was derived from the island of Cyprus in the Mediterranean, where copper was first mined and extracted.

quire complex treatment; in Montana the procedure is as follows: Partial oxidation by roasting and subsequent fusion in Bessemer converter (with silicious lining) sand and air are blown through the molten mass. The iron is oxidized and combines with silica forming a slag which floats on the copper. Sulphur, arsenic and lead are oxidized and volatilized.

Copper is refined by electrolysis in the following manner: thin copper sheets coated with graphite are suspended in tanks of copper sulphate solution and connected with the negative pole of the dynamo, opposite are heavy plates of crude copper connected with the positive pole. Pure copper is deposited on the cathode, while the SO<sub>4</sub> ionizes the anode. The impurities not ionized fall to the bottom of the tank.

**Physical Properties.**—Copper is a red metal melting at 1057° C. and is a good conductor of heat and electricity; it is very malleable and ductile.

Chemical Properties.—Copper slowly oxidizes in dry air forming Cu<sub>2</sub>O and in moist air green basic carbonate (not verdigris). It resists the action of cold dilute acids and alkalies and is much in demand for the manufacture of apparatus used in food preparation, *i.e.*, vacuum pan for sugar, milk, etc., apparatus for canning and preserving, candy making, beer brewing, etc. Large hotels and restaurants use copper cooking utensils.

The most important alloys of copper are:

Brass containing	18—40% Zn, 11% Zn, 3—8% Sn, some Pb
Gun metal containing	10% Sn,
Bell metal containing	25% Sn,
German silver containing	19-44%, Zn, 6-22% Ni.

Metallic copper and its alloys are readily cleaned with dilute oxalic acid or ammonia.

Experiment 1.—Hold a small spiral of copper wire in the upper part of the Bunsen flame until it glows, then immediately drop it into a test tube of methyl alcohol. After the first violent action is over pour out the liquid and observe its odor; of what does it remind you? Examine the wire spiral, note its brilliancy and color. It is clean copper.

**Experiment 2.**—Heat small pieces of clean and tarnished copper separately in the following liquids: dilute HCl, dilute acetic acid, 10 per cent. caustic soda, dilute ammonia. Finally pour off the liquids and add to each an excess of ammonia, a blue color shows the presence of copper. Have the pieces of copper been visibly affected?

**Experiment 3.**—Grasp a short piece of stout copper wire in the fingers and hold the other end in the Bunsen flame, what happens, has this any bearing on the use of copper for cooking utensils?

**Experiment 4.**—Expose small pieces of copper in widemouthed bottles containing a small amount of carbonic

acid water, keeping them covered with watch glasses and standing in a cool place. Examine the greenish coating on the copper and afterwards dissolve it off with dilute acetic acid and add dilute ammonia to the solution. Copper utensils are often coated with metallic tin to prevent the formation of the green coating (basic carbonate of copper).

Silver, Ag, Argentum found native with copper and also as a sulphide chiefly associated with lead sulphide galena; native silver is extracted by electrolysis as described under copper. Silver is separated from the lead extracted from galena (see lead) by the process of zinc desilverization and cupellation. Zinc and lead are quite insoluble in each other and silver is more soluble in zinc than is lead, taking advantage of these facts, the process is operated as follows: silver lead is melted in large cast iron kettles and the zinc added and well stirred. On standing and partially cooling the zinc carrying silver and a little lead, rises and forms a crust which is skimmed and heated in retorts to drive off zinc. The residue Pb+Ag is then heated in a reverberatory furnace with bone ash bed. The lead oxidizes, melts and is absorbed by the bone ash leaving the silver.

In Mexico, the "Patio" process is used. This consists in roasting the ores and treating with copper chlorides to produce AgCl. The silver chloride is reduced and dissolved with mercury forming a liquid alloy (amal-

gam), which is subsequently distilled to free it from mercury.

Silver is a soft white metal, highly ductile and malleable; an excellent conductor of heat and electricity, melts at 960°. It does not oxidize readily in air but is rapidly attacked by sulphides, producing Ag<sub>2</sub>S, (black).

In order to harden silver, it is alloyed with copper in the following proportions: coin silver, 900 parts silver, 100 parts copper. Sterling silver, 925 parts silver, 75 parts copper. All solid household silver is now "Sterling."

Many silver ornaments contain even less silver but articles stamped "sterling" are trustworthy. Silver platedware consists of articles fashioned of German silver or pewter, on which is deposited by electrolysis a triple or quadruple coating of pure silver. The process is similar to copper plating, the silver bath consisting of potassium silver cyanide KAg(CN)<sub>2</sub>. The coating has a frosted appearance and needs burnishing or smoothing before use. Since the coat deposited in this manner is pure silver, these articles do not stand as much careless and rough handling as the harder sterling or coin ware, much of the coating is rubbed off in the process of cleansing with the so-called silver polishes. Plated ware will last much longer if simply washed with hot water and neutral soap. In order to remove the tarnish due to sulphides (eggs), soak the articles in a clean tin dish pan containing enough baking soda solution to cover and



let them remain until bright. The soda solution is made by dissolving a tablespoonful of NaHCO<sub>3</sub> in a quart of tepid water. Silver is insoluble in alkalies, but dissolves readily in strong mineral acids, particularly nitric acid. Oxidized silver is prepared by dipping the object in a solution of hydrogen potassium sulphide KHS until sufficiently blackened.

Mirrors of metallic silver are superior to any others. They may be made as follows: add to a weak solution of silver nitrate just enough ammonia to dissolve the precipitate first formed, then a little caustic soda and finally a little more ammonia and glycerine, glucose, formaldehyde or a neutral tartrate. The glass article to be silvered should previously be cleaned with nitric acid, water and strong alcohol in the order mentioned. The clean object is now immersed in the liquid and gently heated until a sufficient coating is deposited. Try silvering a convex glass or large test tube by this method.

Tin, (Stannum) Sn, occurs in Cornwall and the East Indies as Cassiterite (tin stone) SnO<sub>2</sub>. The ore is crushed and washed to remove rock, roasted to oxidize sulphide of iron and copper and to remove arsenic, then leached with water to dissolve sulphate of iron and copper, dried and reduced with coal in a reverberatory furnace. Tin is a soft silver white crystalline metal, malleable, but not ductile, it melts at 233° C. On bending bar

tin, a peculiar crackling sound is heard called the "cry of tin." Tin is non-poisonous.

Tin plate is made by dipping carefully cleaned sheets of iron or steel in molten tin. It is much used for roofing, household ware and cans for preserving food. Care must always be exercised that tin vessels are not over heated since the element has a low fusion point and will run off leaving the iron bare, therefore it should never be used in the oven or for broiling, roasting or frying. Liquid mixtures may be cooked in tin vessels without doing any damage.

Various useful alloys are known, viz., bronze, soft solder (half tin, half lead) pewter 25 per cent. lead, britannia metal 10 per cent. antimony.

**Experiment 1.**—Heat a small piece of tin plate over the Bunsen flame, note the crystalline appearance on cooling; treat a piece with moderately strong acid and note a similar effect. Where have you frequently seen this phenomenon?

**Experiment 2.**—Scrape some tin from a sheet and heat it with a blowpipe on charcoal before the oxidizing flame, cool, moisten with nitrate of cobalt solution and heat again strongly. Note the dark green mass on cooling.

**Experiment 3.**—Dissolve tin in hot strong HCl, cool and add a little mercuric chloride solution to the liquid; observe the white precipitate turning black.

$$SnCl_2+2HgCl_2=SnCl_4+Hg_2Cl_2$$
.  
 $Hg_2Cl_2+SnCl_2=Hg_2+SnCl_4$ .

Experiment 4.—Join small pieces of tin by "soldering" as follows: Heat the soldering iron (copper) slowly in the Bunsen flame. While heating prepare the tin by rubbing with a clean rag and sprinkling with rosin powder. When the iron is hot, wipe it gently with a cotton rag and bring it directly in contact with the tin at the joint, at the same time touching the end of the iron with the bar of solder. A little of the solder will melt off and may be run up and down the joint until it is closed. If the tin is rusty, scrape it bright and touch with a solution of zinc chloride; apply hot solder and rub with the hot iron until the spot is well soaked.

Brass and copper may be soldered in the same manner. In order to do good work, the point of the soldering iron should always be well tinned. This is done by first filing the hot copper or rubbing it with ammonium chloride, immediately afterward with rosin powder and solder.

Tin plate for roofs and gutters frequently contains much lead.

Test for the lead with bismuth flux on charcoal or plaster plate. Lead, Pb (Plumbum) occurs principally as galena, PbS, (frequently carrying silver). The metal is obtained by roasting the ore until partially converted

into oxide and sulphate. On closing the furnace doors and increasing the heat, the charge is reduced to metal:

PbS+2PbO=3Pb+SO<sub>2</sub>. PbS+PbSO<sub>4</sub>=2Pb+2SO<sub>2</sub>.

Lead is gray in color, soft, of slight tensile strength but very malleable. Melting point 326° C. It is only slightly soluble in acids and alkalies. Lead pipes are formed by forcing warm lead through steel dies by hydraulic pressure. They are much used for conducting water in the household. The danger of drinking water conducted by lead pipes is much exaggerated. Unless the water is unusually soft, the interior of the pipe quickly becomes coated with insoluble sulphate and carbonate. A wise precaution with new plumbing is to allow the water to run for some minutes before use. Lead enters into many useful alloys previously mentioned.

Lead oxidizes superficially, the compound formed being the black suboxide, Pb<sub>2</sub>O, formerly used in place of graphite for lead pencils.

Oxide of lead is very soluble in acids and alkalies.

**Experiment 1.**—Hammer a small piece of lead into a cube, notice how easily it can be formed in any shape (lack of cohesion). Observe the soil on the fingers.

**Experiment 2.**—Heat the small cube prepared in the first experiment in a shallow porcelain or iron crucible, stirring with a stout iron wire, noting the rapidly form-

ing crust of PbO, If the heat is not carried too high, the metal will all turn to oxide in a short time.

Experiment 3.—Divide the oxide formed in Experiment 2 into two equal parts. Dissolve one in dilute HNO<sub>3</sub> and the other in acetic acid; pour a little of each solution in two separate watch glasses and set them aside to evaporate. Examine the crystalline residue in each case. Scrape two pieces of lead bright and immerse one in strong nitric acid, the other in acetic acid; allow them to stand several days and then examine.

**Experiment 4.**—Immerse bright lead in carbonic acid water; after several hours standing pour off the water and test with hydrogen sulphide.

Treat small pieces of bright and tarnished lead separately in weak solutions of acids and alkali. Pour off the clear solutions, acidify with HNO<sub>3</sub>, where necessary, and test for lead by passing H<sub>2</sub>S through the liquid. A black precipitate (PbS) indicates lead.

Each student should make a tabular statement giving the results of the action of acids and alkali on the foregoing metals.

## Chapter VI

### ACIDS AND BASES (ALKALIES)

The most common and useful acids are:

Hydrochloric (HCl), sulphuric (H<sub>2</sub>SO<sub>4</sub>), nitric (HNO<sub>3</sub>) and acetic (HC<sub>2</sub>H<sub>3</sub>O<sub>2</sub>).

Hydrochloric acid (HCl) is the saturated solution of the gas, HCl, in pure water under N. T. P. conditions. It is clear colorless liquid with pungent odor and strong acid taste, sp. gr. 1.2 and contains 40 per cent. of the gas.

Hydrochloric acid is an excellent solvent for metals and insoluble salts.

On heating, the concentrated solution gradually loses gaseous HCl until a sp. gr. of 1.1 (20 per cent.) has been reached; the balance of the liquid then distills of constant composition.

The commercial form known as muriatic acid always contains iron, giving a yellow color to the solution, but may be used for generating H<sub>2</sub>S, etc. For laboratory purposes, the acid is used in the concentrated form sp. gr. 1.2 and in the dilute form sp. gr. 1.1; the latter is prepared by diluting the concentrated acid with an equal volume of distilled water.

Free hydrochloric acid occurs in the gastric juice,

the solution having an average strength of 0.2 per cent. For artificial digestion experiments, this liquid is prepared by adding 6.5 cc. of the concentrated acid to one liter of distilled water.

Combined hydrochloric acid may be identified and determined by means of a solution of silver nitrate with which it makes a curdy white precipitate, soluble in ammonium hydroxide.

Free hydrochloric acid is indicated by a violet or purplish color when heated with a weak solution of egg albumen (see tests on proteins, page 143). A more delicate test (Boas') is made by warming a few drops of the liquid with several cubic centimeters of a solution of 1 gram of resorcin and 3 grams of sugar, in a porcelain dish, if present the HCl develops a red color.

Sulphuric acid, H<sub>2</sub>SO<sub>4</sub>, (HO)<sub>2</sub>SO<sub>2</sub>, a heavy, non-volatile, colorless, viscous liquid, b.p./330, sp. gr. 1.84, containing 96 per cent. H<sub>2</sub>SO<sub>4</sub>. It mixes with water in all proportions, developing great heat during the operation. Caution: In diluting, always pour the cold acid slowly into cold water contained in a beaker or thin flask.

On account of its strong affinity for water, the acid is an excellent drying agent for gases. A small beaker half filled with the conc. acid exposed in the balance case keeps the atmosphere dry for weeks, for the same reason it is used in desiccators. The concentrated acid, especially when heated, is a powerful oxidizing agent, splitting into H<sub>2</sub>O, SO<sub>2</sub> and O (Kjeldahl Digestion). The dilute form acts as a general solvent and hydrolyzing agent. For laboratory purposes, the concentrated and dilute forms sp. gr. 1.1 (H<sub>2</sub>SO<sub>4</sub> 1 vol., water 7 vols.) are used. A special acid (sp. gr. 1.8) is employed in the Babcock process for determining fat in milk.

Commercial sulphuric acid or oil of vitriol has a sp. gr. of 66° Bé. It is brownish in color, due to organic impurities and is also likely to contain arsenic, lead and oxides of nitrogen. This form of acid is extensively employed in manufacturing chemistry, in the production and purification of hydrocarbons, coal tar colors, etc.

Sulphuric acid forms two classes of salts, normal and acid or bi salts, according as both or one hydrogen are replaced.

Combined sulphuric acid (sulphates), if soluble, precipitates a solution of barium chloride forming white crystalline barium sulphate insoluble in acids. Free sulphuric acid carefully evaporated with sugar solution over a water bath chars the liquid.

Nitric acid HNO<sub>3</sub>, HONO<sub>2</sub>, a pungent colorless liquid sp. gr. 1.42 (69 per cent.) soluble in water in all proportions and not volatile at ordinary temperatures.

It is a powerful oxidizing agent, two molecules splitting into H<sub>2</sub>O, 2NO, 3O, forms nitro compounds with organic substances. Ex. nitrocellulose, nitroglycerine, combines with protein substances forming insoluble yellow com-

pounds (acid stains on fingers and clothing). It is used as a general solvent, but unlike other acids does not yield hydrogen when attacked by metals. Concentrated and dilute forms (HNO<sub>3</sub> I vol., H<sub>2</sub>O 2 vols., sp. gr. I.I) are useful laboratory reagents.

Commercial nitric acid is known as Aqua Fortis. It is a yellow liquid, sp. gr. 1.37, containing oxides of nitrogen and other impurities.

For tests on free and combined nitric acid see experiment 11. There is much misconception concerning the strength of the above mentioned mineral acids. According to their equivalents, 36.5 pts. pure HCl, 49 pts. of H<sub>2</sub>SO<sub>4</sub>, and 63 pts. HNO<sub>3</sub> neutralize the same amount of alkali—see Chapter VI. Normal Solutions.

Acetic acid, HC<sub>2</sub>H<sub>3</sub>O<sub>2</sub>, CH<sub>3</sub>COOH is a colorless, strong smelling, sour liquid volatile at 118°.

The usual forms are the 50 per cent. and the glacial acid. Acetic acid is very generally used as a solvent in the laboratory. In the household, a 5 per cent. solution containing extractive matter is known as vinegar. The equivalent of acetic acid is 60.

Commercial acetic acid, a product of wood distillation is largely used in the manufacture of white lead, pigments, dyes, etc. The preparation of vinegar is due to the action of bacteria "mother of vinegar" on dilute alcoholic liquids, beer, light wine, cider, etc. The change is due to oxidation and can only take place in the pres-

ence of air. In the old fashioned practice, oak casks lying on their sides in cool dark cellars were half filled with the alcoholic liquid, the bungs were left out for free access of air, and in the course of several months the liquid soured. From time to time, some vinegar was drawn off and replaced with fresh alcoholic liquid.

The modern method known as the "Quick" vinegar process is operated as follows: large oak casks with perforated bottoms are filled with beech wood shavings and saturated with strong cloudy vinegar, the bacteria collect on the shavings and when the alcoholic liquid is allowed to trickle through the cask, the large oxidizing surface makes the action rapid. The liquid is passed through the cask several times.

Acetic acid forms two classes of salts, metallic and organic or ester; it is monatomic.

The glacial form of acid produced by lowering the temperature until the acid crystallizes, is used as a dehydrating agent. Combined acetic acid (metallic base acetate) is identified by heating with a mixture of sulphuric acid and ethyl alcohol producing ethyl acetate (ester), recognized by its odor (cider).

- I. Experiments on the Acids.—Dilute 10 cc. of conc. HCl to 100 with distilled water, mix well and place in a small distilling flask: see experiments under water. Slowly distill the liquid and test each 10 cc. for acidity.
  - 2. Neutralize 10 cc. of carbonate of soda solution

with HCl, evaporate the liquid to dryness in a porcelain dish, examine the product.

- 3. Test the solubility of marble, gypsum, iron rust and copper in hydrochloric acid.
- 4. Add concentrated HCl to a saturated solution of salt, explain the phenomenon.
- 5. Place 37 cc. of conc. H<sub>2</sub>SO<sub>4</sub> in a 50 cc. flask, add 13 cc. of cold water and mix well. On cooling, how many cc. of water must be added to bring the liquid to the mark?
- 6. Drop a crystal of CuSO<sub>4</sub> into conc. H<sub>2</sub>SO<sub>4</sub>, explain what happens.
- 7. Add dilute sulphuric acid to solutions of barium chloride, calcium hydroxide, lead acetate and silver nitrate. Explain what happens in each case, write equations.
- 8. Half fill two test tubes (6 inch) with dilute nitric acid, add pulverized copper oxide to one and a strip of clean copper foil to the other. Explain the difference of action, write equations.
- 9. Soak a small piece of absorbent cotton for some time in a mixture of conc. nitric and sulphuric acids, take out, wash and dry; is it soluble in alcohol and ether? Try to burn another piece, what happens?
- 10. Treat small pieces of sheet zinc with hot concentrated HNO<sub>3</sub> for several minutes, cool, add excess of NaOH. What gas is evolved, explain?

- 11. Thoroughly moisten starch with strong HNO<sub>3</sub>. What gas is evolved during the action?
- 12. Pour one inch of conc. H<sub>2</sub>SO<sub>4</sub> into a large test tube, add carefully a solution of ferrous sulphate (do not mix); cool if necessary, and add very dilute nitric acid. Note the brown ring. (Test for nitrates).
- 13. Add a few drops of strong nitric acid to some weak albumen; warm gently and note the color (yellow).
- 14. Plunge pieces of bright iron, copper, zinc and lead in separate test tubes containing strong acetic acid. Is there any action after standing some minutes? Finally blow the expired breadth, by means of a glass tube, through the tube containing the copper, what happens?
- 15. Boil about 100 cc. of ordinary 5 per cent. acetic acid in a beaker and from time to time test the vapor with blue litmus paper, when the liquid is low add hot water and boil again, test as before. Can you drive off all the acid at the boiling point (212°)?
- 16. Evaporate 5 cc. of pure acetic acid (50 per cent.) in a clean porcelain dish. Is there any residue?

#### BASES

The most common and useful are the alkalies, soda, potash, ammonia and lime.

Caustic soda, sodium hydroxide NaOH is a deliquescent white solid very soluble in water (212 parts in 100 cc. cold) and develops great heat during solution.

The solid as well as solution absorbs CO<sub>2</sub> from the air, forming sodium carbonate, hence should be kept in closely stoppered bottles. Caustic soda also attacks glass, especially when the surface is roughened, therefore, it is necessary to coat glass stoppers or stopcocks with heavy hydrocarbon when used in contact with this liquid.

Aqueous solutions of 10 and 20 per cent., and an alcoholic solution of four per cent. normal solution, are useful in the laboratory. Caustic soda solutions are used largely in neutralizing acids and in the saponification of fats. For normal solution of caustic soda see end of chapter.

Caustic potash, potassium hydroxide KOH, is similar in physical and chemical properties to caustic soda, slightly less soluble in water (200 parts in 100 cc. cold).

Potash being the weaker and more expensive alkali is now little used in laboratory and commercial operations.

Fats saponified with potash yield very soluble and deliquescent products, "soft soaps" in contrast to the drier sodium compounds, "hard soaps."

For laboratory purposes, the strength of aqueous solutions is the same as for soda.

Ammonia, ammonium hydroxide, aqua ammonia, a saturated water solution of the gas NH<sub>3</sub> in distilled water

under N. T. P. conditions. The solution has a sp. gr. of 0.9 and is diluted with an equal volume of water for general use.

Lime, caustic or quicklime, a dry white solid, is produced by heating carbonate of lime in the form of marble or limestone to a white heat in kilns, the operation being known as "lime burning."  $CaCO_3=CaO+CO_2$ .

Lime has a strong affinity for water undergoing the process of slaking, during which considerable heat is evolved:

If the water is absorbed slowly the heat is given off slowly and is almost imperceptible, the lime masses falling as a soft white powder. Lime also absorbs CO<sub>2</sub>, reconstituting CaCO<sub>3</sub> from which it was derived (air slaking).

Slaked lime dissolves sparingly in water (I part in I200 cc.), forming a clear mildly alkaline liquid known as lime water. A supersaturated solution, milky in appearance, is known as milk of lime (whitewash).

Experiment 1.—Preparation of caustic soda or potash, "causticing." To 100 cc. of a hot 20 per cent. solution of Na<sub>2</sub>CO<sub>3</sub> or K<sub>2</sub>CO<sub>3</sub>, add milk of lime in small portions until a small filtered sample will not effervesce with dilute acid. Boil the remainder of the mixture for 5-

10 minutes and allow it to stand until the white precipitate has completely settled. Draw off the clear liquid with a siphon and keep in a well stoppered bottle; when cold determine the gravity with a Beaumé hydrometer. A 16° solution is the strongest that can be made by this process; stronger solutions are obtained by evaporating the liquid.

**Experiment 2.**—Determine the sp. gr. of various solutions of caustic soda and potash.

Experiment 3.—Neutralize various acid solutions with soda of potash.

Experiment 4.—Boil a small quantity of fat or oil with an equal volume of 16° soda, what is the product?

**Experiment 5.**—Dilute strong ammonia with one, two and four volumes of water, take the gravity of the products. Slowly distill the weaker, what is the distillate, does any of the ammonia remain in the still?

Experiment 6.—Prepare lime by slaking fresh lump lime, allowing it to stand, pouring off the first water containing soluble impurities. Add fresh water and shake well. The second clear liquid is pure lime water, taste, expose some to air of the room and blow the expired breath through another portion. How much lime water does it take to neutralize 10 cc. of 0.2 per cent. hydrochloric acid, to neutralize 5 cc. of ordinary vinegar, and the same quantity of sour milk whey?

### NORMAL SOLUTIONS OF ACID AND ALKALI

A normal solution is one which contains the hydrogen equivalent of substance in grams, in one liter of solution. For all monobasic acids and alkalies the hydrogen equivalent corresponds with the molecular weight of the compounds; for dibasic substances it is one-half of the molecular weight. In similar manner tri- and tetrabasic bodies have hydrogen equivalents corresponding to one-third and one-quarter of their molecular weight.

Normal solutions may be made of one-tenth or onehundredth of their full strength, either by taking the corresponding fractions of their respective equivalents or by diluting the full normal solutions proportionately; they are known as deci- and centinormal solutions respectively.

To explain the preparation of the normal solutions of acid and alkali, one example from each class will suffice and as hydrochloric acid and caustic soda have the most extensive application, their preparation will be given. Neither the acid nor the alkali can be weighed or measured with accuracy, hence it is first necessary to make up solutions of some acid or alkali which can be made exact. Sodium carbonate, whose equivalent is 53, can be obtained of a high degree of purity and may be weighed exactly. It is hardly necessary to make up a large quantity, so that 5.3 grams of pure dry soda are

usually weighed accurately, dissolved in the least quantity of water and the resulting solution diluted to exactly 100 cc. at or about 60° F. This constitutes the exact normal soda, I cc. of which contains 5.3 milligrams of soda.

Of the hydrochloric acid, 36.5 grams are needed but as it is a volatile liquid and cannot be weighed with any accuracy, it is usual to calculate the volume of the liquid from its specific gravity and weight, and to measure out the result in cubic centimeters, allowing a little for loss. The calculation is simple and is made as follows: divide the equivalent in grams (36.5) by the specific gravity of the concentrated acid (1.2); this gives 30.4 + as a quotient and is the number of cubic centimeters to be used if the acid were pure, but the strongest acid is only 40 per cent., hence this quotient must be multiplied by  $2.5 (30.4 \times 2.5 = 76 \text{ cc.})$ . It is safe to take 78-80 cc., adding it to 300 or 400 cc. of distilled water and when cool diluting to exactly one liter. To fix the strength exactly and make it equivalent to the soda solution proceed as follows: Measure 10 cc. of the soda very exactly with a pipette, run it into a small beaker containing about 100 cc. of distilled water, and add two or three drops of methyl orange solution. a burette with the acid solution. Note the level, and run it, drop by drop, with constant stirring, into the soda; stop when the last drop changes the color from vellow

to pink which remains even after stirring for some moments. Read the burette and note the number of cubic centimeters, and fractions used. Say the quantity is 9.8 cc., indicating that this quantity contains as much acid as should exist in 10 cc.; consequently, 980 cc. of the liquid should be diluted to one liter. If the total amount of acid is less, calculate what bulk it should occupy and dilute accordingly. The acid keeps very well but should be preserved in tightly stoppered glass bottles to prevent evaporation.

The caustic soda is deliquescent and absorbs carbon dioxide so must be weighed rapidly and approximately, using rather more than the 40 grams required, say 50 grams. This is dissolved in 300 or 400 cc. of water, cooled and diluted to one liter. Draw off 10 cc. of the normal acid in a pipette, allow it to run into a small beaker containing about 100 cc. of distilled water, and add a few drops of phenolphthalein. Fill a clean, dry burette with the caustic soda. Note its level and run it, drop by drop, with constant stirring, into the acid solution until a faint but distinct pink tint remains after stirring for some moments. Read off the quantity used, say 9.5 cc., showing the solution to be too strong and requiring dilution as in the case of the acid. After performing this operation the acid and the alkali should be correct and I cc. of one will exactly neutralize an equal quantity of the other.

To test unknown substances, first determine the body present by qualitative analysis, and then weigh or measure some convenient quantity, dissolve or dilute with distilled water, add the indicator and run in the acid or the alkali until the neutral point is reached. Observe the number of cubic centimeters used and multiply each by its value in milligrams of the substance sought and divide the result by the quantity used; multiplying this quotient by 100 will yield per cent.

Value of I cc. of normal soda in each of the following:

Sodium Carbonate	0.053
Acetic Acid	0.060
Lactic "	0.090
Tartaric "	0.075
Citric "	0.064
Hydrochloric Acid	0.0365
Nitric Acid	c.063
Sulphuric Acid	0.049
Potassium Hydroxide	0.056
Ammonium "	0.035
Calcium "	0.037

## Chapter VII

### GLASS, POTTERY, AND PORCELAIN

These substances belong to a series of infusible and insoluble silicates of great utility in all household operations. Glass consists of a mixture of silicates in the amorphous state and is highly prized on account of its brilliancy and transparency: the mass may be colored without affecting either of these qualities. The usual varieties of glass consist of a mixture of alkaline (with alkaline earth) or heavy metal silicates, and are known as Bohemian, Crown, Bottle and Flint glasses.

Bohemian glass is a silicate of potash and lime. It is very infusible and insoluble, therefore especially adapted for chemical purposes. Two varieties, hard and soft, are mentioned in the introductory chapter.

Window or Crown glass is a silicate of soda and lime. It is more fusible but harder than the Bohemian and is more easily affected by acids.

Bottle glass is an impure variety of the above, colored with iron.

Flint glass is a potash lead silicate. This is the most fusible kind of glass and is easily attached by chemical reagents; on account of its high refractive power, it is much used for optical purposes.

All kinds of glass are prepared by fusing more or less pure silica in the form of sand or powdered quartz with the potash or soda and lime or red lead, for many hours in large earthenware pots, heated in appropriate furnaces. When the mass has cleared, it is cast or blown and cooled rapidly in order to retain its transparency.

Annealing is a process of heating to a temperature short of softening and cooling slowly, thereby reducing the brittleness.

While transparency is a very important property of all glasses, there are several useful opaque forms. Opaque glass is the result of suspending finely divided infusible material in the molten mass. Such materials are bone phosphate, cryolite, zinc or tin oxides, etc. The enamels used on cooking utensils are of similar composition.

One of the most characteristic properties of all glasses is the solvent effect of hydrofluoric acid and soluble fluorides. Etching on glass is largely accomplished by this means.

Colored glass is the result of dissolving some appropriate mineral oxide in either variety of glass:

Ruby—oxide of gold or copper.

Topaz—sulphide of antimony.

Yellow-silver chloride or borate.

Green-oxide of chromium.

Blue-oxide of cobalt.

Amethyst-oxide of manganese.

#### **EXPERIMENTS**

- r. Effect of Sudden Cooling.—Heat glass tubing strongly in the bunsen flame until it softens and immediately plunge into water. Note the effect. This change will take place even in cold dry air or sudden contact with a cold surface.
- 2. Effect of Slow Cooling.—Heat as before, but finally coat with carbon by cutting off the air supply to the burner. When cool enough to handle, wipe off the carbon with clean dry filter paper, and note that the glass has not changed.
- 3. Corrosive Action of Alkalies.—Half fill common prescription bottles (4 oz.) with strong caustic soda solution. Place them in warm salt water, bring slowly to a boil and continue for at least one hour, then cool slowly, pour out the contents, rinse with clean water and examine the inner surface.
- **4. Etching Tests.**—(a) With a clean steel pen and dilute HF, write your name and the date on a clean microscope slide.
- (b) Thinly cover a clean watch glass with warm paraffin. When cool cut your name with a pencil point through the paraffin and immediately invert over a lead dish containing a mixture of fluorspar and concentrated sulphuric acid. After half an hour's standing, rub off the paraffin and examine the result.

5. Detection of Arsenic, Lead, Etc.—Fuse finely ground chips of kitchen utensil enamel with an excess of potassium sodium carbonate in an iron or nickel crucible, cool and extract the melt with hot water. Filter, and wash the residue several times with hot water. Test the filtrate for arsenic, lead, and acids by dividing it into three parts—two of one quarter each and the third the remaining half.

Part I. Test for arsenic by making strongly acid with HCl and boiling with a strip of clean copper. A gray or black coating indicates arsenic.

Part II. Make acid with HCl and pass H<sub>2</sub>S rapidly through the solution. A black precipitate indicates lead

Part III. One-half of the solution—tests for sulphates, borates, phosphates, and silicates, as follows:

Neutralize with HCl, if any precipitate forms, filter and divide the filtrate into three parts. The residue is silicates. Take one part of the filtrate, thoroughly moisten a strip of turmeric paper with it and dry at 100° C. A pink color indicates borates.

To another part, add barium chloride and a few drops of HCl. A white crystalline precipitate indicates sulphates. Pour a few drops of the remaining part into an excess of ammonium molybdate. Warm gently and a yellow color or yellow crystalline precipitate indicates phosphate.

Porcelain and Pottery are fused silicates of alumina, the former pure, and the latter contaminated with oxides of iron, manganese, etc.

The primary source of these wares is clay, a hydrated silicate of alumina, highly infusible. For porcelain making it is mixed with some fusible silicate such as feldspar, and a small quantity of water, moulded into shape, dried and heated in a furnace for many hours. The feldspar or flux only melts and running through the porous mass cements it together. Even after firing, the ware requires coating with the glaze, a mixture of slightly fusible material suspended in water into which the article is dipped. It is dried and returned to the furnace for heating. The glaze is, in effect, a true glass and makes the mass impenetrable to liquids. Decorative effects are produced in two ways; called under- and overglaze, of which the former is the best and most permanent. For under-glaze work, finely ground colored glass suspended in turpentine is applied to the unglazed ware and afterwards "fired" at the high temperature of the porcelain furnace. The glaze is subsequently applied and fired as before. Over-glaze decoration admits of the use of colors which may be injured by the high heat of the porcelain furnace and is applied at a lower temperature in a muffle. The colors consist of various oxides mixed with borax, litharge, nitre, etc. They are applied in watery solution.

Stoneware is an impure form of porcelain, somewhat more fusible and usually glazed with borax. The finer qualities are known as china. Earthenware and brick consist of clay and sand, mixed with water, moulded, dried and fired in a kiln, the former is usually glazed with salt, while the latter is left in the porous state.

#### **EXPERIMENTS**

- 1. Compare the relative translucency, hardness, and brittleness of porcelain, china, and earthenware.
- 2. Testing the Glaze.—Boil small pieces of decorated china in caustic soda or carbonate of soda solutions (10 per cent.) for some time (1 hour). Cool, wash and compare with untreated pieces.
- 3. Porosity.—Weigh small pieces of dry unglazed porcelain and earthenware, soak over night in water, wipe dry and weigh again. Calculate the per cent. of water absorbed.
- 4. Fusibility.—Heat small splinters of porcelain and earthenware held in platinum wire (spiral) at the highest heat of your burner. Cool and examine with a magnifier. Are the edges sharp or rounded?
- 5. Testing for Lead in the Glaze.—Boil the article for some time in caustic soda, cool the liquid and add  $(NH_4)_2S$ . A darkening of the liquid or a black precipitate indicates lead.

## Chapter VIII

### PAINTS AND VARNISHES

These substances serve primarily as a protective coating, for wood, metal and masonry surfaces; in a secondary sense, they are used for decorative effect.

Paint consists of some amorphous solid called the base, suspended in a liquid known as the vehicle. Any insoluble coloring matter may be added for tinting pur-Two kinds of paint are commonly used; viz., water, and oil. The simplest water paint is whitewash or milk of lime, with or without salt or plaster to increase the adhesive quality. Whitewash loses water (dries) and absorbs CO<sub>2</sub>, turning to chalk, CaCO<sub>3</sub>. Kalsomine is finely ground chalk suspended in a warm dilute solution of glue. It is applied warm and dries and hardens on cooling. Any appropriate coloring matter may be mixed with either of the above. Since these mixtures are largely compounded of water, they will not be permanent when exposed to moisture or dampness; hence their use is confined to the interior of buildings. Mixtures of boiled soap and lime or skimmed milk and lime, usually classed as whitewashes, become so insoluble on exposure to air, that they are superior in lasting quality to any except the best oil paints.

Oil paints contain as base, either white lead (basic

carbonate of lead, Pb(OH)2, PbCO3) or zinc white (ZnO) or a mixture of both, suspended in the vehicle, linseed or poppy seed oils. When mixed, the base and cil form soaps, soluble in the excess of oil present, but not in water. During the drying operation, the oil absorbs oxygen and solidifies to a tough elastic body. This latter action is however quite slow, hence driers (compounds of manganese) are used to hasten the absorption of atmospheric oxygen. Spirits of turpentine (C<sub>10</sub>H<sub>18</sub>) commonly called "Turps" are useful in thinning the paint mass and causing it to penetrate the freshly painted surface. Turps also dulls or flats the otherwise glistening surface of the dried paint, but renders it at the same time noticeably susceptible to the action of moisture. As in the case of water paints, mineral colors ground in oil are used for tinting. The following is a list of the most lasting:

Reds-Indian, Venetian, Tuscan (oxides of iron).

Yellows—Ochre (oxide of iron), Chrome (chromate of lead).

Brown-Umber (manganese clay) Sienna.

Blue-Prussian Blue.

Green-Prussian Blue and Chrome Yellow.

Black-Lamp- or Bone-black (carbon).

Oil paints are best applied in at least three successive coats. First or priming coat is pure white lead and oil with abundance of turps. This coat sinks in and dries

rapidly. Second or body coat is pure zinc and lead base, linseed oil and drier. It is much thicker than the previous coat and takes longer to harden and dry. Finish coat is much like the body coat but thinner; in rare cases it may contain a little turps for flattening effect.

Varnishes are of two kinds-spirit and oil.

Spirit varnishes consist of gum resins dissolved in alcohols; for instance, shellac and the less common lacquers. As the solvent evaporates, the gum remains as a continuous coat. Because of their light color and transparency, they are much used in covering wood-work. Shellac is tender and spots with water; hence its use is limited to furniture, metals and interior wood-work.

Oil varnishes are prepared by dissolving fossil resins in hot linseed oil, an operation requiring much skill and time. After ageing for several months they are ready for use. Many varieties receive an addition of turps before use, especially if used for interior work. A good oil varnish will outlast even the best paint and if applied as a finishing coat will increase its life many times. Carriages, railway coaches and automobiles are painted in this manner.

It is a good plan never to dilute any varnish of standard make if you expect the best results.

#### **EXPERIMENTS**

1. Testing Water Paints.—Evaporate a small portion

in a porcelain dish. As the mass dries, observe the odor. Does it suggest glue? Does the residue blacken, indicating organic matter? Ignite until white or nearly so, cool and add dilute acetic acid. Any effervescence is probably due to  $CO_2$ , indicating carbonates. Is the mass entirely soluble in acetic acid; if not pour off the liquid, test it for lime and add dilute HCl to the residue. Heat this for some time, finally driving off the acid, cool and add water. Is the residue gritty, indicating silica, SiO<sub>2</sub>?

- 2. Testing Oil Paints—Preferably White.—Half fill a clean 4 oz. vial with gasoline, add about one teaspoonful of the sample, cork and shake well. Lay it aside to settle, then pour off the clear liquid, add more gasoline to the vial and shake again. Repeat this once more, finally couring the whole contents into a shallow dish or saucer and drive off the remaining solvent over hot water. Carefully evaporate a portion of the gasoline filtrates and observe the thick oily residue. Add to it two drops of H<sub>2</sub>SO<sub>4</sub> conc.; does it form brown rings where the acid drops fall (indication of linseed oil)? The dried residue, freed from gasoline, should dissolve in dilute acetic acid (freedom from clay, barites, etc.). Pass H<sub>2</sub>S through the solution and filter off the black precipitate of PbS. To the filtrate add NH4OH cautiously; a white precipitate indicates Zn.
  - 3. Testing Varnishes.—Little or nothing can be done in

a simple way to identify the gums, but some indication of the solvent may be obtained by the following method:

Distill a small quantity of the varnish and examine the distillate for alcohol, turpentine and benzine. The odor is usually sufficient indication; also note the temperature of distillation.

# Part II

## Chapter IX

Food Principles.—They are types of chemical compounds that serve to maintain heat, muscular energy and repair work of organisms. Carbohydrates, fats, proteins and mineral salts are the accepted forms.

Carbohydrates.—A series of compounds of the alcohol and aldehyde type, containing C, H and O. The H and O atoms in the ratio of 2:1 and the C, six or some multiple. Familiar examples of these compounds are starch, cellulose, sugar, glucose, etc.

The carbohydrates are divided into three groups:

- 1. Monosaccharids—glucoses, C<sub>6</sub>H<sub>12</sub>O<sub>6</sub>.
- 2. Disaccharids—sugars,  $C_{12}H_{22}O_{11}$ .
- 3. Polysaccharids—celluloses and starches, nC<sub>6</sub>H<sub>10</sub>O<sub>5</sub>.

Both poly- and disaccharids are convertible into monosaccharids by the process of hydrolysis, which consists in the absorption of H and O as they exist in water; this process may be carried out in a variety of ways, two of which acid and enzyme hydrolysis are most important.

Ultimate Composition.—1. Determination of Hydrogen and Oxygen Evolved as Water.—Heat about 1 gram of

granulated sugar in a clean dry test-tube. Observe the condensation of moisture in the cooler part of the tube.

- 2. Determination of Carbon. (a) By direct heat, continue heating and note the blackening of the mass due to freeing of some of the carbon. (b) By dehydration. Treat about I gram of the sugar in a porcelain evaporating dish with a little concentrated sulphuric acid, heat gently and note the blackening effect. In this case, the concentrated sulphuric acid withdraws the hydrogen and oxygen in the form of water and leaves the carbon.
- 3. Determination of Hydrogen and Carbon in the Form of Hydrocarbons, i.e. Tarry Bodies.—Observe the pungent odor of the vapors arising from the tube during the heating in experiment 2, (a). This usually condenses on the cooler part of the tube in the form of a dark brown liquid (caramel).

Starch, celluloses and dextrines are the most common and important forms of polysaccharids.

STARCH  $(C_6H_{10}O_5)_n$ 

Starch, a complex plant product is formed in the leaf from atmospheric moisture and carbon dioxide, through the agency of sunlight and chlorophyl according to the following:

 $H_2O+CO_2=HCOH+\hat{O}_2$  by condensation. 6 $HCOH=C_6H_{12}O_6$  by dehydration.  $C_6H_{12}O_6=C_6H_{10}O_5+H_2O$ . The formula,  $C_6H_{10}O_5$ , does not express the ultimate composition of starch, but merely the ratio in which the constituents always occur. The actual formula is some large multiple of this simple form, which serves however for all practical purposes.

Starch thus formed is stored up by the plant in minute particles known as granules, varying in size and shape. The larger and simpler forms are found in the roots, the smaller in the seeds. There is no known chemical difference in these various forms.

In general terms raw starch consists of a mass of unbroken, and cooked starch of broken granules.

Raw starch is a white opaque solid, neutral in reaction, insoluble in cold water, alcohol and ether. Heated to 250° F. it begins to decompose darkening in color, giving off an odor of burning wood and acid vapor; at higher temperatures, it chars and leaves a porous mass of carbon. If starch is held in suspension in water and gradually heated at 160° F., it begins to gelatinize due to bursting of the granules; at or about 212° this change takes place rapidly.

A solution of iodine in potassium iodide and water, colors raw or cooked starch blue, provided the mass is cool and either neutral or acid: the reaction is due to the formation of iodide of starch of indefinite composition. Gelatinized starch is slightly soluble in cold water.

By hydrolysis starch changes slowly into dextrines, maltose and glucose (dextrose) as follows:

$$\begin{array}{l} 2C_{6}H_{10}O_{5} + H_{2}O = C_{12}H_{22}O_{11}. \\ C_{12}H_{22}O_{11} + H_{2}O = 2C_{6}H_{12}O_{6}. \end{array}$$

Two per cent, of mineral acid H<sub>2</sub>SO<sub>4</sub> or HCl is the amount usually employed, slight pressure during boiling, greatly shortens the operation.

One-tenth of one per cent. or less of enzyme (diastase) at 150-160° F. rapidly converts starch into dextrine and maltose, but the final stage must be completed by acid.

Starch is rapidly soluble in concentrated sulphuric acid (hydrolysis). It unites with concentrated nitric acid in varying proportions, forming nitrates, very unstable compounds (explosives).

In presence of cold strong fixed alkali, starch is soluble with partial hydrolysis and usually the product has a distinct yellow color; weaker solutions have very little effect unless heated.

#### **EXPERIMENTS**

## ORDINARY STARCH DERIVED FROM POTATOES OR CORN

1. Occurrence of Starch.—Examine a thin section of potato under the microscope. Make a careful drawing of the structure of the cells and the granules within. Cover the section with a thin glass and introduce a minute trace of iodine solution at the edge of the cover

glass. Note and make a colored (blue pencil) diagram of the result.

- 2. Extraction of Starch.—Clean and peel one end of a small potato, rub it on an ordinary grater, collect the gratings in a beaker of cold water, strain, allow the cloudy liquid to stand until starch settles. Pour off liquid and use the sediment for following tests:
- 3. Effect of Dry Heat.—Gently heat half an inch of dry starch in a clean, dry test-tube observe and explain condensed moisture in the cooler part of the tube. Increase the heat somewhat and note the odor of the evolved vapor and the color of the starch: what does it suggest? Now heat strongly until only a black residue remains: what is it?
- 4. Effect of Strong Acid.—To a small portion of dry starch in a porcelain evaporating dish add a few drops of concentrated sulphuric acid; note the result and after a short time heat gently and observe again.
- 5. Solubility.—Treat a small portion of finely pulverized dry starch with cold water, filter a portion and examine the filtrate for dissolved material, by evaporating a little on platinum foil: also by the iodine test.
- 6. Soluble Starch.—Boil the remainder of the starch and water mixture: it gelatinizes. Filter some of this and test the clear, cold filtrate with iodine. Explain. To the remainder when cool add a minute portion of

iodine solution: it is colored blue. Gently heat this and allow to cool again: note the result. Now boil for some time, cool, the color will not return. Examine under the microscope portions of raw and cooked starch, with and without iodine.

- 7. Effect of Alkali.—To some starch solution in a testtube add a small portion of caustic soda (NaOH) and a few drops of iodine solution and note the result. Repeat the experiment using dilute sulphuric acid instead of NaOH.
- 8. Effect of Tannic Acid.—Add a solution of tannic acid to some starch solution. Note the result, also any change effected by heating.
- 9. Starch, a Colloidal Substance.—Prepare a dialyzer as directed and partly fill it with starch solution, then stand the whole in a beaker of cold water. After standing for some time test the water for starch with iodine solution.
- 10. Hydrolysis.—Make a very weak solution of starch in about four ounces of boiling water: to this solution add 2 cc. of strong hydrochloric acid and boil until clear, using a reflux condenser. At this point, a small quantity of the cooled liquid should give no blue coloration with iodine solution. If this is not the case add 10 drops of the same acid and boil some minutes longer, or until a small portion gives no test with iodine as above. Now neutralize the remainder of the liquid with sodium carbonate solution.

- ing 5 cc. of copper sulphate and alkaline Rochelle salt (the same amount) with 20 cc. of distilled water, boil the solution exactly two minutes. During this period no change should take place in the liquid. Add I or 2 cc. of the neutralized starch solution and boil again for two minutes. Note the change in color of the liquid and when cool, the red precipitate of cuprous oxide, Cu<sub>2</sub>O, indicating the presence of a reducing sugar, *i.e.* glucose. If the indicated changes do not take place add more solution and boil again.
- 12. Enzyme Hydrolysis.—Unorganized ferments: ptyalın from saliva or diastase from malt. Prepare saliva in the following way: rinse out the mouth with water, curve the tongue so as to place its tip behind the upper incisor teeth, then inhale the vapor of ether or even cold air: collect the saliva in a small test-tube, dilute with five times its volume of water and filter through a filter paper perforated with a point of a pin. The filtered liquid should react neutral or alkaline.

Make a starch solution in hot water, cool, add a few cc. of saliva, warm (not above 40° C.) until clear, cool and test with (a) iodine solution, and (b) Fehling's solution (maltose).

To a weak starch solution add about 5 cc. of diastase solution and heat to 65° C. As soon as the paste becomes clear, test a portion with iodine solution and con-

tinue testing other portions until the test fails to give a color (maltose). At this stage boil the remainder of the solution with about 25 drops of dilute sulphuric acid for ten minutes. Neutralize and test this with Fehling's solution (dextrose).

# $\begin{aligned} \mathbf{DEXTRINE} \left\{ \begin{array}{l} \mathbf{Erythrodextrine} \\ \mathbf{Achrodextrine} \\ \mathbf{Maltodextrine} \end{array} \right. & (\mathbf{C_6H_{10}O_5})_x \end{aligned}$

**Preparation.**—Dextrine or "British Gum" is prepared commercially by heating starch moistened with nitric acid. It may be prepared more conveniently by heating a strong starch paste with moderately dilute sulphuric acid, cooling and precipitating the dextrine by adding to ethyl alcohol.

**Solubility.**—Compare the solubility of dextrine in cold water and in boiling water.

To successive portions of cooled dextrine solution in test-tubes add:

- 1. Iodine solution.
- 2. Alcohol up to 60 per cent. by volume.
- 3. Caustic potash and iodine solutions.
- 4. Sulphuric acid and iodine solutions.
- 5. A few drops of ammonia and basic acetate of lead: note the result. This is the characteristic test for gums.
- 6. To boiling Fehling's solution: if pure there will be no reaction.

- 7. Tannic acid as under starch.
- 8. Take about 25 cc. of clear dilute starch solution in a small beaker and add 2 or 3 cc. of undiluted saliva, keep at body temperature and from time to time pour off small portions and test with iodine solution, keeping each for comparison. Note the gradual change from blue to red to yellow and finally to colorless. The stages are: starch, blue: erythrodextrine, violet: achrodextrine, yellow: maltodextrine, colorless.

## GLYCOGEN (C<sub>6</sub>H<sub>10</sub>O<sub>5</sub>)<sub>x</sub>

Preparation.—Grind a mixture of scallops and sand in a mortar, transfer to a beaker, add enough water to cover the mass and boil. This dissolves the glycogen and partially precipitates the proteins, which are now completely precipitated by adding a few drops of acetic acid. Filter and to the filtrate add alcohol (95 per cent.) until glycogen comes down as a white precipitate. Allow to settle, decant off the clear liquid, and filter the remainder.

Apply the following tests to the glycogen thus obtained:

- 1. Solubility in water; look for opalescence.
- 2. Solubility in 10 per cent. sodium chloride solution.
- 3. Solubility in hydrochloric acid.
- 4. Solubility in caustic potash.
- 5. Reaction with iodine solution.

6. Boil in a beaker for fifteen minutes with 2 cc. of dilute hydrochloric acid, neutralize with sodium carbonate and test with Fehling's solution. What change has taken place?

Glycogen is soluble in alcohol from 0-60 per cent. and insoluble in alcohol from 60-100 per cent.

## CELLULOSES (C<sub>6</sub>H<sub>10</sub>O<sub>5</sub>),

These compounds represented by the general formula  $nC_6H_{10}O_{15}$  are at once the most complicated and stable of the carbohydrates.

They may be roughly divided into the simple and compound celluloses, the former unicellular in structure, and the latter multicellular.

Cotton, thistledown and the internal fibrous network of grains and vegetables are simple celluloses and occur as ribbon-like bands with curled edges and a characteristic corkscrew twist. These forms contain little protein, gum, fat or mineral matter. Flax, grasses and woody fiber are compound celluloses occurring for the most part, as jointed rods or tubes, and are highly charged with protein, fat, gum and mineral matter. Cotton is the only unicellular form of carbohydrate of industrial importance, while the multicellular type has many representatives, *i.e.*, linen, hemp, jute, ramie and a great variety of woods.

The treatment of cotton does not involve any exten-

sive chemical operations, but is chiefly confined to mechanical manipulation. The compound celluloses on the other hand require complex and prolonged chemical or bacterial treatment before the fiber is ready for the operations of spinning, weaving and dyeing. Woody fiber is now generally used for the preparation of the felted fabric known as paper. It is necessary in this case to remove all impurities by chemical means, and to break up the long fibers by grinding before the fabric can be prepared.

General Properties of the Celluloses.—Celluloses are insoluble in water hot or cold, and in weak acids or alkalies. Strong acids and alkalies cause them to hydrolyze; in some cases soluble forms resulting by heating or prolonged action, in the cold or by a combination of both methods. Generally speaking, the action of acids is more rapid. When partially hydrolyzed they are colored blue in the presence of iodine. Nitric acid in concentrated form converts cellulose into nitro compounds of varying composition, containing one to six nitro groups; the form depending on the duration of the nitrating process. All of these compounds are very unstable and dissociate into water, carbon dioxide and nitrogen, when slightly heated; hence their use as explosives. Nitrocelluloses, unlike cellulose, dissolve in ether, alcohol or acetone or mixtures of these solvents (collodion) and on evaporation yield transparent structureless films; used in medicine, photography and for the preparation of artificial silk. Ammonio cupric oxide (Schweitzer's Reagent) and concentrated zinc chloride dissolve simple cellulose on gentle warming. Hydrocellulose precipitates from these solutions on acidifying with acetic acid.

Lignocellulose (wood) yields oxalic acid on treatment with nitric acid, and oxalate of potash on fusion with

caustic potash.

Cellulose fibers of both types do not readily unite with dyes, but require previous chemical treatment (Mordanting) to develop the color and make it permanent.

Cellulose fibers are characterized by high capillary capacity and heat conductivity; hence their use for lamp wicks, toweling and summer clothing. These properties, however, may be much modified by high twisting and close weaving, as in the case of canvas.

## GENERAL TESTS

- (a) Effect of Heat (Charring).—Heat a piece of fibrous material in a clean dry test-tube. Note the odor of the gases evolved and test the vapor with blue litmus paper. Examine the charred mass with a magnifier.
- (b) Solubility in Water.—Try to dissolve some fibrous material in water.
- (c) Solubility in Concentrated Sulphuric Acid.—Add concentrated sulphuric acid to some fiber. Note the effect.

- (d) Structure.—Examine carefully the structure of the fibers under a microscope.
- (e) Crude Fiber.—Crude cellulose is determined as follows:

Take one gram of the dried, ground sample, boil with 100 cc. of 1½ per cent. sulphuric acid, when cool strain through muslin. Wash once with hot water. Scrape the residue from the muslin and boil it with 100 cc. of 1½ per cent. caustic soda. Strain again through the same piece of muslin, wash with hot water, then with alcohol, and finally with ether. Weigh the dried residue.

- (f) Methods for Determining the Character of Fibers.
- —(1) If a mixed cotton-linen fabric be dipped for about one minute in cold concentrated H<sub>2</sub>SO<sub>4</sub> quickly removed and washed, the cotton fibers alone will be dissolved.
- (2) If a mixed cotton-linen fabric be dipped in a neutralized solution of fuchsine and subsequently washed, the cotton will fail to take the color, while the linen will be dyed pink.

## TESTS ON COTTON

- (a) **Structure.**—Note the ribbon-like structure of pure cotton fiber.
- (b) Breaking and Burning Tests.—Unravelled threads of cotton fabric are untwisted and broken by holding between the thumbs and index fingers and pulled apart

slowly and steadily. Cotton breaks suddenly with tasselled ends. Burn a small tuft of cotton and note the condition of the fiber ends.

- (c) Nitrating.—Treat a piece of filter paper (cotton) with concentrated nitric and sulphuric acids, keeping the mixture cool. Several nitrates of cellulose may form. The hexa- and penta-nitrates are the most prominent. The hexa-nitrate of cellulose is called gun cotton. Wash the product in water and dry. Test its inflammability and its solubility in a mixture of equal parts of alcohol and ether.
- (d) Mercerization.—Soak some cotton cloth in 10 per cent. caustic soda and note that it becomes partially transparent—mercerized. If the cloth is thoroughly washed free from alkali and tested with iodine solution, a blue color appears.
- (e) Solubility in Zinc Chloride.—Dissolve some absorbent cotton in acid zinc chloride solution, (ZnCl<sub>2</sub> dissolved in twice its weight of conc. HCl). Precipitate by dilution and compare the result with the original substance.
- (f) Solubility in Schweitzer's Reagent.—Dissolve some absorbent cotton in Schweitzer's reagent, add the resulting solution to 95 per cent. alcohol and compare the precipitate with the original substance.

#### TESTS ON LINEN

- (a) Structure.—Examine carefully the structure of single linen fibers.
- (b) Breaking and Burning Tests.—Unravelled threads of linen fabric are untwisted and broken by holding between the thumbs and index fingers and pulled apart slowly and steadily. Linen parts slowly, and with pointed ends.

Burn a piece of linen fabric and note the condition of the fiber ends.

(c) Solubility in Concentrated Sulphuric Acid.—Treat a piece of linen cloth with concentrated sulphuric acid and compare the effect with the effect on cotton cloth.

#### TESTS ON LIGNOCELLULOSE

- (a) **Structure.**—Examine carefully the character of the fibers.
- (b) Phloroglucinol Test.—Phloroglucinol, in HCl, gives a deep magenta coloration with any of the lignocelluloses.

The reagent is prepared by dissolving the phenol to saturation in HCl (1.06 sp. gr.).

(c) Saturate moist jute fiber, held in a glass tube, with chlorine gas and then pass SO<sub>2</sub> through it. Note the characteristic reaction a deep magenta color.

#### TESTS ON PAPER

Determine starch as filler with iodine solution. De-

termine "size" with Millon's reagent. If protein material is present a white precipitate, turning red on heating forms.

Parchment Test.—Dip starch-free paper in a cold mixture of water and sulphuric acid (2:3), withdraw quickly, wash in clear water and dry. Compare with an untreated sample. Does it respond to the iodine test, why?

## GLUCOSES (C<sub>6</sub>H<sub>12</sub>O<sub>6</sub>)

These bodies are now regarded as aldehydes of the type CH<sub>2</sub>OH(CHOH)<sub>4</sub>COH. The most common representatives are known as dextrose or grape sugar, levulose or fruit sugar and galactose. A mixture of equal parts, by weight, of dextrose and levulose is commonly called invert sugar. The essential difference in these compounds is due to the effect and degree of their action on polarized light. Dextrose causes a right hand deviation, levulose a left hand, galactose a right hand; but each in turn varies in degree, levulose being greater than either dextrose or galactose.

Glucoses are very soluble in water and more soluble in alcohol than other carbohydrates. As aldehydes they reduce alkaline solutions of metallic salts, to the metallic state or some low form of oxide (see silver mirror test page 66, and Fehling's reaction page 102). All, however,

in the same quantitative proportions. The osazones are formed by the phenylhydrazine reaction.

Glucoses are formed during the acid hydrolysis of other carbohydrates as follows: Cellulose, starch and maltose yield dextrose exclusively; lactose yields a mixture of dextrose and galactose; sucrose yields a mixture of dextrose and levulose. Since sucrose is dextro-rotatory and on hydrolysis yields a mixture of dextrose and levulose of levo-rotatory character, the process is called "inversion" and the product "invert sugar." Such is not the case with other carbohydrates, whose products remain dextro-rotatory.

The tendency to crystallize is not so well marked in glucoses as in the sugars, but is not entirely absent. Dextrose and galactose crystallize readily from ordinary alcohol, while levulose crystallizes with difficulty from absolute alcohol.

Dextrose ferments readily with yeasts and produces for the most part alcohol and carbon dioxide as follows:  $C_6H_{12}O_6{=}2CO_2{+}2C_2H_{15}OH$ . Levulose is less readily fermented but yields the same products. In the case of invert sugar, yeasts attack for the most part the dextrose. Galactose in the pure state is not attached by yeast but when mixed with dextrose, it slowly ferments. The mixture of dextrose and galactose, obtained by hydrolyzing milk sugar, is most susceptible to the action of

lactic acid bacteria; the following equations explain the change:

$$\begin{split} &C_{12}H_{22}O_{11},\ H_{2}O{=}2C_{6}H_{12}O_{6}.\\ &C_{6}H_{12}O_{6}{=}2C_{3}H_{6}O_{3}. \end{split}$$

#### EXPERIMENTS ON DEXTROSE

Note the taste and roughly determine the solubility in hot and in cold water. Does it react with iodine solution?

- r. Effect of Heat.—Heat some dry dextrose in a clean dry test-tube, note the result.
- 2. Effect of Strong Acid.—To some dry dextrose in a porcelain dish add cold concentrated sulphuric acid: note the result. After allowing the test to stand for five minutes, heat gently and again note the result.
- 3. Effect of Strong Alkali.—To some dextrose solution add strong caustic soda or potash and heat: note the result.
- 4. Crystallization.—Make a syrupy solution of dextrose and allow it to stand for several days. Do any crystals form? Add an equal volume of 95 per cent. alcohol, mix well, let stand twenty-four hours. What result?
- 5. Fermentation.—Rub up some dextrose solution with a small piece of compressed yeast in a porcelain mortar. Pour the mixture into a short broad test-tube until it is full of the liquid. Close the test-tube with a perforated cork, bearing a glass tube in the form of the letter J.

The short end of the tube should pass through the cork into the broad test-tube, while the long end should reach to the bottom of a test-tube containing lime water. Set the apparatus away in a warm place, and allow it to stand for twenty-four hours. Why does the lime water become cloudy? Examine the liquid in the broad tube for alcohol by taste, and by heating with a few drops of iodine and sodium carbonate solution—the odor is that of iodoform.

- 6. Silver Mirror Test.—See page 66.
- 7. Fehling's Solution Test.—In a 100 cc. flask take 5 cc. of copper sulphate solution and 5 cc. of alkaline Rochelle salt, mix and add 20 cc. of distilled water, and a few pieces of pumice stone to prevent bumping, and boil for two minutes. No change should take place. Add a few drops of dextrose solution, boil vigorously for two minutes, cool and note the result. Continue adding dextrose and boiling until on cooling the blue color of the solution has faded and a red precipitate is formed. Compare with Experiment 10 under starch. It requires 0.050 gram of dextrose to reduce the Fehling's solution.
- 8. Test weak dextrose solution with Nylander's reagent under the same conditions as observed with Fehling's Test. Note the result.
- 9. Test weak dextrose solution with Barfoed's reagent heating over boiling water. Note the result.

10. To 5 cc. of dextrose solution in a test-tube add 0.1 gram of phenylhydrazine hydrochloride and 0.2 gram of sodium acetate. Heat the mixture gently until all solids dissolve, then keep it in boiling water for fifteen minutes. Cool and examine the yellow radiating needles of phenylglucosazone.

Examination of food materials (flours, cereals, bread, crackers, etc.) for starch, dextrine and dextrose.

Make a cold water solution of the finely ground material to be tested; allow to stand for several minutes, filter through starch-free paper. Test the residue for insoluble starch, with iodine. Test the filtrate (1) for soluble starch and dextrine (a) with iodine. (b) With alcohol. (2) For dextrose with Fehling's Test.

# SUGARS $(C_{12}H_{22}O_{11})$

The disaccharids or sugars of the general formula,  $C_{12}H_{22}O_{11}$ , include such well known substances as sucrose, maltose and lactose. Unlike the glucoses they are not identical in composition. Sucrose for instance is an alcohol with no trace of aldehyde or ketone character as shown by its behavior toward Fehling's reagent, while maltose and lactose possess these characteristics reducing the reagent in marked degree.

The sugars are readily soluble in water and crystallize with ease; facts which are utilized in their isolation. On the other hand they hydrolyze rapidly at low temperatures

with all acids, hence must be protected by neutralizing.  $C_{12}H_{22}O_{11}+H_{2}O=2C_{6}H_{12}O_{6}$ .

Plants accumulate their maximum sugar content at a period just short of maturity. If the sap is decidedly acid, little sugar will be found in the mellow stage, the change to the invert form being almost complete.

# SUCROSE $(C_{12}H_{22}O_{11})$

Sucrose is identical whether separated from cane, beets or maple juice.

#### **EXPERIMENTS**

- 1. Examine the crystalline structure of granulated sugar.
- 2. Roughly determine its solubility in cold water. Is the solubility affected by heat?
- 3. Effect of Dry Heat.—Boil down sugar solution to dryness and note the result.
- 4. Effect of Strong Acid.—Drop some concentrated sulphuric acid on dry sugar, note the result and compare with starch and glucose.
- 5. Effect of Alcohol.—Add a saturated solution of sugar to 95 per cent. alcohol and note the result.
- 6. Boil weak sugar solution with Fehling's solution. If pure there should be no reaction.
- 7. Hydrolysis "Inversion."—Boil sugar solution with a few drops of concentrated hydrochloric acid, cool, neutralize with sodium carbonate solution and heat again

with Fehling's solution: note the result and compare with glucose. What change has taken place?

- 8. Test sugar solution with Nylander's reagent, before and after boiling with acid.
- 9. Repeat test with Barfoed's reagent as in Experiment 9, under glucose.
- 10. Effect of Strong Alkali.—To 10 cc. of sugar solution add some strong caustic potash, heat and note the result: compare with glucose.
- II. Crystallization.—Make a hot syrupy solution of sugar and suspend in it a piece of glass rod by thread. Set aside and allow to cool and after a time carefully examine the crystals of cane-sugar.
- 12. Specific Test.—To 15 cc. of the clear liquid, add 5 cc. of cobalt nitrate (5 per cent.) and 2 cc. of caustic soda (50 per cent.). Sucrose gives an amethyst-violet, permanent on heating. Dextrose gives a turquoise-blue, turning to green on heating. This test may be used on milk, preserves, etc. To remove gum or dextrine, add ammonia and basic acetate of lead, filter and test the filtrate.
- 13. Caramel Test.—Boil a strong solution of sugar until it has turned brown (caramel), cool, dilute and test some of the liquid with Fehling's solution.
- 14. Saccharimeter Test.—Certain substances when dissolved possess the power of rotating the plane of po-

larized light, some to the right, others to the left. Cane sugar is dextro-rotatory. For the saccharimeter test (using the Ventzke scale) 16.37 grams of cane sugar are dissolved in exactly 100 cc. of distilled water. The tube of the instrument is then filled with the solution and the percentage of sugar read directly on the graduated scale of the instrument.

After making the first reading, dilute some of the solution with an equal volume of water and make another reading on this solution.

For the saccharimeter test, the solution must be clear and colorless. In case it is not, the color may be discharged by means of a solution of basic acetate of lead, which precipitates dextrine and gummy matter, and by bone-black, which removes soluble coloring matter. The operation is conducted as follows:-16.37 grams of sugar are dissolved in somewhat less than 100 cc. water, a few cc. (not more than five) of the lead solution are added and the solution diluted to mark. The mixture is shaken vigorously and then allowed to stand for a few minutes for the bulky precipitate to set-If perfectly clear and colorless, the liquid is poured into the tube; if the color has not entirely been removed a subsequent treatment with bone-black is nec-For this purpose bone-black which has been dried at a red heat is mixed with the solution and stirred for some minutes. The mixture is then filtered through

dry paper, the first 10 cc. of the filtrate being rejected. It may be necessary to refilter the liquid through the same paper two or three times.

## MALTOSE $(C_{12}H_{22}O_{11})$

Maltose does not occur in nature, but is produced during the hydrolysis of starch by unorganized ferments, such as diastase, ptyalin, etc.

Preparation of Malt.—Malt is produced during the germination of barley and other cereals. Prepare it as follows: spread out a thin layer of barley grains (one tablespoonful) on the cover of a small pasteboard box, moisten with warm water and keep in a moderately warm place. Each grain will soon begin to sprout. When the rootlet has grown the length of the grain, dry it in an oven at a low temperature and keep bottled.

Make malt extract by grinding the grains coarsely and extracting them with 100 cc. of warm water. Note the taste and odor of the liquid. Keep for future use.

**Preparation of Pure Maltose.**—Make a thin paste of starch and boiling water, cool to 65° and add 10 cc. of malt extract, prepared as above, and continue the heating at 65° C. for half an hour. From time to time test small portions of the liquid with iodine solution when the liquid fails to react blue, cool the balance of the solution, divide in parts and test as follows:

1. Solubility in Alcohol.—Add some of the liquids to

strong alcohol, allow to stand and note the white precipitate of dextrine: the liquid contains maltose.

- 2. Effect of Fehling's Solution.—To 10 cc. of Fehling's solution add a few drops of the liquid, boil for two minutes and note the reduction: add more of the solution and boil again: repeat until the reduction is complete. The solution is reduced by 0.08 gram maltose.
- 3. Effect of Barfoed's Reagent.—Test with Barfoed's reagent.
- 4. Effect of Nylander's Reagent.—Test with Nylander's reagent.
  - 5. Apply the fermentation test.
  - 6. Test with strong caustic soda or potash.
- 7. Repeat the phenylhydrazine test. Compare with glucose.

## LACTOSE $(C_{12}H_{22}O_{11}, H_2O)$

Method of Preparation.—Allow milk to stand until it has become decidedly sour, filter. Heat the whey to coagulate the albumen, filter again. Evaporate the filtrate over hot water, to crystallization. The crystals are lactose.

- 1. Note the hardness and slightly sweet taste, due to the limited solubility.
  - 2. Try its solubility in water and in alcohol.
- 3. Treat some dry powdered lactose with concentrated sulphuric, note the result.

- 4. Try the caustic potash reaction.
- 5. Apply the Fehling's test. It requires 67 mgs. to reduce the solution.
  - 6. Test with Barfoed's reagent.
  - 7. Test with Nylander's reagent.
- 8. Make a weak solution of lactose in water, let it stand at least twenty-four hours in a moderately warm place, and then test for acidity.
- 9. Make the phenylhydrazine test, compare with glucose and maltose.



# Chapter X

## METHODS FOR TESTING FLOURS, MEALS, ETC.

Samples to be tested: viz., white bread, baker's, macaroni and whole wheat flour.

Examine small portions of each under the microscope as in lesson under starch. Make paste, stain with iodine and examine again.

1. Preparation of Gluten.—Take 25 grams of the sample, mix on a porcelain or glass plate with the least amount of water to make a stiff dough (12-15 cc.). Do not handle the dough with the fingers, use a flexible steel knife. Transfer the dough to a well-washed piece of muslin, taking care to clean the mixing surface and knife thoroughly; tie up the muslin in the form of a bag and wash under a gentle stream of water, manipulating well with the fingers. Continue the washing until the liquid runs clear from the bag, and fails to give the test for starch with iodine. Be careful to collect all the washings in a tall glass beaker or jar-(they should amount to from one to one and a half liters). Strain through muslin and stand the filtrate aside in a cool place for settling. Reserve for future use. Examine the residue with a lens. Squeeze out as much water as possible from the bag, untie it and collect and weigh the

moist gluten; spread it out in a thin cake and dry it for one hour at 100°-105° C. Cool and weigh the dry gluten.

2. Tests on Protein and Soluble Carbohydrates.—When the contents of the jar (used in last experiment) has settled, decant off the clear liquid, and test small portions of it for protein with the Biuret test and for soluble carbohydrate with Fehling's reagent.

Note. Biuret Test—To I in. of 10 per cent. NaOH or KOH, add very dilute CuSO<sub>4</sub> solution, drop by drop, until a faint blue color, but no precipitate appears in the liquid. Now add the protein solution—a violet color indicates protein, a pink peptone.

3. Tests on Starch.—Pour distilled water on the residue in the jar and stir up the mixture, allow it to settle and decant as before. Repeat this operation twice, and then collect the residue on balanced filter-papers; dry and weigh. This last weight gives the starch and fiber content but in the case of ordinary wheat flours the latter is so small that it may be neglected.

Note—In the case of whole wheat flour, it is best to pass the sample through a 100-mesh sieve, taking the screenings for the determination of starch, gluten, etc.

4. Determination of Ash.—Incinerate about 5 grams of the sample in a porcelain dish, cool, and moisten the ash with a few drops of concentrated nitric acid. Add water, boil and filter. Test the filtrate (1) for po-

tassium with platinic chloride (2) for phosphoric acid with ammonium molybdate.

Rye Flour.—Examine under the microscope as under wheat flour. Take 25 grams of rye flour and pass it successively through screens of 40, 60, 80 and 100-mesh; weigh and examine the residue retained by each sieve and also test them for starch with iodine. Treat the material which has passed the 100-mesh sieve for the determination of starch, gluten, etc., as described under wheat flour.

Ash a small portion and determine the mineral constituents as under wheat flours.

Corn Meal (Yellow).—Examine under the microscope as before. Test a small portion in a corked test-tube with ether or benzine; shake vigorously and when the upper layer of liquid is clear, decant it through a filter-paper and cautiously evaporate the liquid in a clean, dry evaporating dish. Note the character of the residue; What is it?

Treat about 25 grams for the determination of starch, protein, etc., as before.

Make ash determination as before.

Experiments on Raw Potatoes.—I. Select a small sound potato, clean well and carefully grate it over a shallow tin dish. Allow a thin stream of water to play upon the grater during the operation; not more than one quart

of water should be used. Pour the gratings and liquid through a muslin strainer into a tall glass jar and allow the contents to settle. Examine some of the material on the filter with a lens, and test it with iodine.

- 2. When the contents of the jar have settled, draw off some of the clear liquid and test for protein and soluble carbohydrate.
  - 3. Test the settlings for starch.

Note.—To preserve these, wash several times with dilute salt solution, drain and dry at a low temperature.

Experiments on Cooked Potatoes.—1. Take a small well baked potato. Remove and dry the skin, grind in a mill and test the clear water solution and the insoluble residue for starch, protein and mineral matter.

- 2. Examine the white interior of the potato for uncooked starch grains, with a microscope.
- 3. Make a solution of a portion of this material, filter and subject to the same tests as used on the skin.

Experiments on Bread.—Separate the crumb and crust.

Crust.—Grind the darker portion to a coarse powder, add water and mix thoroughly. Add more water, boil the mass for some time, cool and filter. Divide the filtrate into four parts and test as follows:

- I. Add iodine solution and note the result.
- 2. Observe the taste; explain.
- 3. Add to Fehling's solution and boil; note the result.

4. Pour a few drops into strong alcohol.

Crust.—Test the crumb as follows:

- 1. Add iodine solution and note the result.
- 2. Make test for insoluble protein (gluten). Which test is best?
- 3. Burn some crumb to a gray ash in a porcelain dish, cool, digest ash with warm nitric acid and divide into three parts. Test one part for chlorides, another for phosphates and the remainder for potassium.

**Experiments on Toast.**—Grind toast to powder, treat one teaspoonful (level) with boiling water for five minutes, filter hot, cool. Divide liquid into four parts and test as follows:

- 1. With iodine solution.
- 2. With Fehling's solution.
- 3. With tannic acid.
- 4. Add a few drops of the cooled liquid to strong alcohol.

Experiments on "Cereals or Breakfast Foods."—This classification includes the various commercial preparations of oats, corn, barley, wheat and rice, or mixtures of the same. They are supposed to have undergone some operation of cooking and claim to contain no raw starch. The following general tests will serve to give some idea as to the condition of the material and the presence or absence of the various food principles:

- 1. Powder the material as finely as possible in a mortar or coffee-mill. Pass the grindings through a 100-mesh sieve and examine the screenings and siftings separately under the microscope. Stain with iodine and examine for the presence of unbroken starch grains.
- 2. Extract a portion of both screenings and siftings with cold water, filter and examine the filtrates separately for dextrine—(by precipitation with alcohol and color test with iodine), for dextrose or reducing sugars (with Fehling's solution), for maltose (with phenylhydrazine).
- 3. Test portions of the insoluble residue separately for protein.
- 4. Mineral Matter.—Incinerate a fresh portion of the screenings in a porcelain dish, cool and extract the mass with water, filtering and testing the filtrate for chlorides, sulphates and phosphates of potassium and sodium. Test the insoluble residue with a little concentrated nitric acid and boil. Cool, dilute with water, filter if necessary and test the clear liquid for phosphates and for calcium.

# Chapter XI

#### FATS AND OILS

There is much confusion in regard to the composition of the various bodies commonly known as fats.

At least two distinct classes are recognized, viz.:

(1) True fats or glycerides, containing carbon, hydrogen and oxygen, essentially in the form of fatty acid and glycerine, and existing as solids and liquids.

(2) Hydrocarbons containing only carbon and hydrogen in varied forms of combination, but existing as gases, liquids and solids.

On heating the first always yield some of their carbon in the free state, the latter rarely.

Ordinary fats consist of mixtures of at least three glycerides; oleine, liquid at ordinary temperatures, palmitine semi-solid and stearine solid. In very few instances other glycerides are present, notably butter, cocoanut oil, cotton oil, etc. Hence olive oil is liquid because of the preponderance of oleine and tallow hard from excess of stearine.

In fact these mixtures are separable by heating or cooling them to a temperature just short of solidification or liquefaction and pressing the mass.

Fats and oils are insoluble in hot and cold water; solu-

ble in hot alcohol and in cold and hot ether, chloroform and gasoline.

The radicle glyceryl  $(C_3H_{t_5})^{\prime\prime\prime}$  is common to all fats, the fatty acid may vary.

In the process of saponification (fat splitting) glycerine C<sub>3</sub>H<sub>5</sub>(OH)<sub>3</sub> is therefore a constant product.

 $(C_{17}H_{35}COO)_3C_3H_5 + 3HOH = 3C_{17}H_{35}COOH + C_3H_5(OH)_8$ , ultimate composition of glycerides.

Determination of Hydrogen and Oxygen in the Form of Water.—Boil 20-25 drops of clear olive oil in a clean dry test-tube. Note the watery deposit in the cooler part of the tube; some of this running back will cause the fat to crackle.

**Determination of Glycerine.**—Continue heating the tube until dense fumes arise from the boiling liquid. This is due to acrolein, C<sub>3</sub>H<sub>4</sub>O, a decomposition product of glycerine.

 $C_3H_8O_3=C_3H_4O+2H_2O.$ 

Cool the tube and contents and reserve for next step.

Determination of Carbon and Hydrogen as Hydrocarbons Resulting from the Break Down of the Fatty Acids.

—Pour the cold tube contents into a clean dry porcelain dish and heat slowly but strongly over a low flame. Note the gradual darkening of the liquid due to freeing of carbon and the tarry coat on the rim of the dish (hydrocarbons). Hold a lighted match over the dish and note

the inflammable character of the vapor (hydrocarbon gases). Extinguish the flame and continue the heating until only a black residue remains. This is carbon: prove it by burning off.

Extraction of Pure Fat from Animal Sources.—Weigh out 10 grams of beef suet cut up in small pieces. Place in a small evaporating dish and heat cautiously, stir with a thermometer and do not allow the temperature to rise above 130° C. (What causes the spattering?) When the spattering has ceased, strain through muslin into a porcelain dish, squeeze out the cloth and reserve contents for tests on fats.

Transfer the residue to a small mortar, add 5 cc. of strong alcohol, grind well. Pour this mixture into a small flask, wash out the mortar with alcohol and add the washings to the flask. Finally close the flask with a cork, bearing a condenser tube 24 inches long, support on a ring stand over a water-bath and boil for ten minutes. When the suspended matter has settled, uncork the flask and pour the clear liquid on a small filter, allowing the filtrate to run into a large test-tube. To the residue in a flask, add 20 cc. of ether, insert the cork and condenser and cautiously heat over hot water for five minutes. Then transfer the entire contents of the flask to a small muslin filter and collect the filtrate in the same tube as before. Wash this last residue with a little ether and squeeze out the ether, spread out on the

muslin and allow it to dry. Test the residue for protein with Millons's reagent. Close the test-tube with a loose cotton plug, allow it to stand until crystals deposit from the liquid. Examine these under the microscope and draw a diagram of them.

Extraction of Pure Fat from Seeds or Nuts.—Dry and grind the nuts to a fine powder. Place the ground mass in a small Erlenmeyer flask, cover with the solvent (ether), cork and shake well. Remove the cork and heat very gently over warm water. Finally pour the clear liquid through a dry filter into a clean porcelain dish and cautiously drive off the solvent. The clear oily residue is used for tests.

Make the following tests on the rendered (extracted) fat:

- I. **Solubility.**—Test the solubility of small portions of fat in separate test tubes with: water hot and cold, with alcohol hot and cold. Record the results.
- 2. Acidity (Rancidity).—Place a small piece of fat in a clean, dry test-tube; add 5 cc. of alcohol and heat until the fat melts, cool and test the liquid with delicate litmus paper.
- 3. Absorption.—Place a small piece of fat on a filter-paper and heat until the fat melts; note the result.
- 4. Formation of Acrolein.—Rub up a small piece of fat in a mortar with some acid potassium sulphate, trans-

fer to a clean, dry test-tube and heat cautiously; note the peculiar disagreeable odor of acrolein due to the dehydration of the glycerine. What does it suggest?

- 5. Solubility in Na<sub>2</sub>CO<sub>3</sub>.—Warm a small piece of fat in a test-tube with strong Na<sub>2</sub>CO<sub>3</sub>, shake well, noting the result. Allow the mixture to stand: what happens?
- 6. Saponification with Alkali.—To about I gram of fat in a low flask, add 25 cc. of alcoholic potash solution, cover with a watch glass and boil. Replace the liquid lost by evaporation with alcohol. As the heating progresses, the mixture should become homogeneous: if it does not, add a little more potash and boil until clear (saponified). Remove the cover and evaporate the bulk of the alcohol, finally adding hot water and heating until all alcoholic odor has disappeared. Cool the liquid and divide into three parts.
- 7. Precipitation and Decomposition of Soap.—To one portion add a saturated solution of salt; notice the curdy precipitate (soap). Filter off this precipitate, dissolve it in fresh water and reserve for future use. Boil down some of the filtrate, note any change in color or odor and finally, when quite concentrated pour a few drops of the thick liquid on a platinum foil, add bisulphate of potassium, evaporate to dryness and ignite gently. Note the odor and blackening; what does it suggest? (Odor of frying fat.) What are the white crystals?

- 8. Separation of Fatty Acids.—Acidify another portion of the dissolved soap with dilute sulphuric acid. Note the curdy precipitate (fatty acids) which is insoluble in water but soluble in warm alcohol. Boil the mixture until clear, filter and use in test 9.
- 9. Test the solubility of the fatty acids with alcohol and with sodium carbonate solution. Record results.
- ro. Formation of Lime Soap.—Add a solution of limewater to another portion of the soap solution and notice the greasy precipitate, which is insoluble in warm water and alcohol (lime soap, produced by hard water).
- of cod-liver oil and dilute sodium carbonate. Notice the resulting white mass which is called an emulsion; what well known liquid is similar in appearance? Examine two or three drops of this emulsion under the microscope and note the character of the compound.

Repeat the same experiment, using a few drops of olive oil and a solution of albumen.

- 12. Formation of Lead Soap.—Heat a small quantity of lard or tallow, for some time in an evaporating dish, with lead oxide PbO, and a little water; filter off the watery liquid and evaporate a small quantity on a platinum foil. Note the characteristic odor of acrolein, proving the presence of glycerine.
- 13. Melting Points (Lard, Olive or Cottonseed Oil, Tallow).—Determine the melting-point by filling capillary

tubes with the respective liquid fats. Chill them with cracked ice and salt, and fasten to the bulb of a chemical thermometer with a small rubber band. Immerse this system in a beaker of cool water and gradually raise the temperature (not faster than 2° C. per minute). When the contents of the tubes become translucent, the melting point has been reached. Record results in each case.

- 14. Special Tests for Drying Oils.—(a) Becchi's Test.

  To 5 cc. of the oil in a 4-inch test-tube, add an equal volume of silver nitrate dissolved in alcohol (1 per cent. solution); close the test-tube with a cotton plug and keep it in boiling water for ten to fifteen minutes. A darkening of the mixture indicates cotton-seed oil.
- (b) Halphen's Test.—To 5 cc. of the oil in a 6-inch test-tube add 5 cc. of amyl alcohol and 5 cc. of carbon disulphide containing a little free sulphur. Place the tube in a beaker of cold water and bring the water to a boil; allow the tube to remain for twenty minutes. A red coloration indicates cotton-seed oil. This is a very delicate test.
- (c) Drying Test.—Expose separate drops of olive and cotton-seed oils on a microscope slide to a warm dry atmosphere for 12-24 hours. Cool and note any difference in character.

## PREPARATION OF COLD-MADE SOAP

Lye.—Dissolve the contents of one can of Babbett's potash (1 lb.) in one quart of cold water. This gives a solution of about 40° Bé.

Fat.—Tallow rendered according to directions given on page 130. Heat the fat until it is just liquid and add slowly, with constant stirring, lye equal in amount to one-half the fat taken. Stir it thoroughly until homogeneous and pour into a shallow pasteboard box. Allow it to stand for at least twenty-four hours, and then test for free fat and free alkali as follows:

For Free Fat.—Shake a few shavings of the soap in a corked test-tube with cold gasoline, filter into a convex glass and evaporate off the gasoline over warm water. A greasy residue indicates unsaponified fat.

For Free Alkali.—Shake a few shavings of the soap in a corked test-tube with warm alcohol (95 per cent.), filter and add to the clear liquid a few drops of phenolphthalein; a red color indicates free alkali.

#### BUTTER. SPECIFIC TESTS

Melt about a teaspoonful of butter with ten times the volume of warm water, stir the mixture vigorously and then chill by standing it in ice water. Punch two holes in the cake of solid fat and decant off the liquid. Preserve some of this liquid for a test. Taste a portion; test with litmus paper. To a small portion of the solu-

tion add a few drops of silver nitrate and note the result. Continue the washing operation two or three times until the final filtrate is quite clear. Note any difference between the first and last filtrates in respect to taste, and test with litmus and silver nitrate. Carefully dry some of the chilled fat between layers of filter-paper. Melt the fat in hot water and filter it through dry paper. Preserve this fat for future work.

Now wash the paper with a very small portion of cold gasoline until a drop of the washings evaporated on paper leaves no greasy stain, dry and note the character of the residue on the filter-paper. Cautiously heat some of the residue (curd) in a test-tube with lime; observe the odor produced and hold in the fumes a piece of moistened red litmus paper and note the result.

Place a drop of the butter-fat first on water and then on 95 per cent. alcohol; note whether it floats or sinks in either liquid. Mix a small portion of the fat with potassium or sodium bisulphate, heat on a platinum foil, and note the peculiar disagreeable odor (acrolein). Compare the odor of this compound with the odor produced by treating glycerine in the same way.

Spoon Test.—Heat a piece of butter about the size of a cherry in a tablespoon. If it froths without spattering, it is pure butter. If it foams and spatters it is renovated butter: if it spatters only, it is oleomargarine or some other artificial butter.

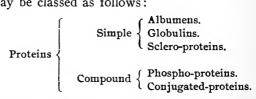
Butyric Acid Test.—In a 4 oz. narrow neck flask, fitted with a one-holed rubber-stopper, put about 2½ grams of butter. Saponify with caustic potash. Decompose the resulting product with dilute sulphuric acid in excess. Then distill the product gently, using a bent tube condenser. Butyric acid will distill at about the temperature of boiling water. Allow the distillate to drop into a funnel containing moist filter paper. This causes the retention of fatty acids (other than butyric). Below the funnel is placed an Erlenmeyer flask containing N/Io alkali, with phenolphthalein as indicator. The excess of alkali, after the distillation is complete, is titrated with N/Io HCl.

## Chapter XII

#### PROTEIN BODIES

These compounds contain carbon, hydrogen, oxygen and nitrogen, sometimes sulphur, phosphorus, iron, lime, etc.

They may be classed as follows:



The ultimate composition of the proteins may be determined as follows:

- I. Determination of Nitrogen as Ammonia.—Mix some dried egg albumen with lime and moisten sufficiently to roll into small balls with the fingers. Place two or three of these balls in a dry test-tube, heat and hold in the vapors a piece of moistened red litmus paper. Note the result. Let the paper dry and observe the change.
- 2. Determination of Sulphur as Hydrogen Sulphide.— Test the fumes with a piece of filter paper moistened with lead acetate and note the result.

- 3. Determination of Hydrogen and Oxygen as Water.— Observe the condensation of water in the cooler part of the tube.
- 4. **Determination of Carbon.**—Observe the blackening effect produced by the freeing of the carbon.
- 5. Determination of Phosphorus.—Moisten the residue from test 4 with concentrated HNO<sub>3</sub> and heat gently until excess of acid has been vaporized, then heat strongly until the carbon has been entirely consumed. Cool the residue, moisten with HNO<sub>3</sub>, add water, boil and filter if necessary. Test the clear liquid with ammonium molybdate.

#### GENERAL PROPERTIES

1. **Solubility.**—Albumens and gelatine are soluble in warm water, other forms are insoluble.

All proteins are soluble in dilute sodium chloride, albumens alone are soluble in saturated solutions.

All proteins are insoluble in saturated solutions of ammonium sulphate.

Make a table based on these facts.

- 2. Coagulation.—Albumens and globulins are made insoluble by heating their neutral or faintly acid solutions to about 70° F.
- 3. Curdling.—Phospho- and conjugated proteins are made insoluble, without serious chemical change, by adding dilute acid.

- 4. Clotting.—Certain enzymes (rennin) split phosphoand conjugated proteins, yielding nuclein and simple protein.
  - 5. All proteins are insoluble in alcohol.
  - 6. Proteins are indiffusible.
- 7. Proteins hydrolyze and yield the following products; meta-protein (acid or alkali), proteose, peptone and polypeptides (amino acids).

For the purpose of making general and specific tests on the proteins, a solution of egg albumen prepared according to the following directions is recommended.

Preparation of Egg Albumen.—Carefully break a fresh egg, allow the clear white to run into a porcelain dish and set the yolk aside for future use. Cut the white with scissors or grind with sand and place a small portion in a wide-mouthed stoppered bottle, add ten volumes of distilled water, shake until it froths and invert over a small casserole of water. When the froth and insoluble protein particles float on the surface, carefully withdraw the cork and allow some of the liquid to mix with the water in the casserole. The liquid will probably be opalescent, due to traces of globulin; if strongly so filter through cloth, test the fluid with litmus paper and if alkaline neutralize with weak acetic acid (2 per cent.).

I. General Tests.—(a) Nitric Acid.—To a small por-

tion of the filtered liquid, add strong nitric acid. This forms a white precipitate which turns yellow on heating; now cool and add ammonia—it becomes orange. Compare with spots on the skin or woolen cloth produced with HNO<sub>3</sub>.

- (b) Biuret Test.—To I inch of 10 per cent. caustic soda or potash, add dilute copper sulphate, drop by drop, until a faint blue color but no precipitate remains in the liquid after shaking; now add the protein solution. A violet color indicates protein; a pink, peptone.
- (c) Precipitation Tests.—Solutions of the proteins are precipitated by the following reagents:

Alcohol.

Tannic Acid.

Picric Acid.

- (d) Coagulation by Heat.—Heat some of the fluid to boiling and add, drop by drop, very dilute acetic acid (2 per cent.) as long as a precipitate forms; note that this precipitate does not appear unless the solution is acid. Attempt to filter some of the albumen through a wet filter-paper; prove by one of the above tests that no protein is in the filtrate. Repeat the above test, using first undiluted egg albumen and second a very dilute solution (1 cc. to 100 cc. of water).
- 2. Special Tests for Albumens and Globulins.—(a) Millon's.—To a small portion of the solution, add Millon's

reagent and heat. This forms a white precipitate which turns red on cooling, or gives a red color if only a trace of protein is present.

- (b) Heller's Test.—Place some strong nitric acid in a test-tube and allow a solution of albumen to flow gently down the sides of the tube; a white ring of precipitated albumen forms at the junction.
- (c) Metaphosphoric Acid Test.—Add a solution of albumen to a very little cold freshly prepared metaphosphoric acid and note the precipitate formed.
- (d) Precipitation Tests.—To portions of the solution in separate test-tubes add:

Acetic acid and potassium ferrocyanide.

Mercuric chloride.

Lead acetate.

- 3. Separation Tests.—(a) To a portion of the solution, add an excess of dry crystallized ammonium sulphate, shake vigorously. Albumen and globulin will be precipitated, without change in composition.
- (b) To a portion of the solution, add dry sodium chloride or magnesium sulphate. Globulin, only will be precipitated.
- 4. Indiffusibility.—Place some of the solution in a dialyzer of parchment paper and suspend the whole in a beaker of distilled water. Test the water subsequently

for chlorides with silver nitrate and also for protein by the biuret test.

- 5. Proteolysis.—(a) Acid Metaprotein.—To undiluted egg white add conc. HCl; note the copious precipitate of albumen (coagulated). Heat gently until the mass dissolves resulting in a violet solution. Cool and dilute some of this liquid testing it as follows:
  - 1. Heat to coagulating point (70° C.).
  - 2. Exactly neutralize with dilute caustic soda.
  - 3. Make the biuret test.
- 4. Add a few drops to 15-20 cc. saturated sodium chloride.
  - 5. Add a few drops to 15-20 cc. 95 per cent. alcohol.
- (b) Alkali Metaprotein.—Treat undiluted white of egg with strong alkali; note the clear jelly-like mass which results. Dilute some of this with water and make the following tests:
  - 1. Heat to coagulating point (70° C.).
  - 2. Exactly neutralize with dilute acetic acid.
  - 3. Make the biuret test.
- 4. Add a few drops to 15-20 cc. saturated sodium chloride.
  - 5. Add a few drops to 15-20 cc. 95 per cent. alcohol.

Note. Weaker solutions of albumen are converted by treating with a few cc. of very weak alkali or acid (0.1 per cent.) at 100° F., for some minutes.

(c) Preteose and Peptone.—The action of pepsin is hydrolytic and produces both proteose and peptone—a case similar to the production of dextrine and dextrose from starch.

Preparation of Preteose and Peptone.—Coagulate egg albumen by heat. Cut into small wedge shaped pieces, put into three test-tubes and treat as follows:

- I. Cover with highly dilute hydrochloric acid (0.2 per cent.).
- 2. Add water and a very small amount of neutralized pepsin solution.
  - 3. Add both pepsin and hydrochloric acid.

Place all three tubes in a beaker of cold water, heat to body temperature and note the time they take to clear: also observe whether the mass swells: finally filter all three and test the clear filtrates for peptone by the biuret test.

#### GLOBULINS

Globulin from the White of Egg.—Saturate some of the undiluted solution with dry magnesium sulphate, grinding the mass in a mortar. Observe the precipitate of globulin, filter and test the filtrate for protein, now pour water through the insoluble mass on the filter and test the extract for proteins. Explain. The yield of globulins obtained from this source is very small and the following method is preferable.

Globulin from Hemp-Seed.—Extract dry, ground hemp-seed with 5 per cent. salt solution, heating the mixture gently but not above 50° C.; filter and test the clear filtrate as follows:

- 1. Heat to coagulating point: what is it?
- 2. Add sodium chloride to saturation, filter and test the precipitate with nitric acid and with biuret.
- 3. Make the biuret test for protein on some of the saline solution.

#### YOLK OF EGG

Place one-half the yolk of a fresh egg in a broad sixinch test-tube, add twice its bulk of ether, cork and shake well: allow the tube to rest until the contents separate into layers, uncork the tube and pour carefully the upper (ether) layer into a clean porcelain dish and reserve for future use. Add another portion of ether to the tube, cork and shake. Allow it to stand until it has settled, pour off the clear ether solution as before, adding it to the ether solution contained by the first operation. Repeat these washings at least four times or until the residue in the tube is white or nearly so. Dry the residue over hot water and reserve for future test, it is principally vitellin.

Evaporate the combined extracts over hot water (no flame). When the ether has passed off, note the yellow liquid oil similar to melted butter.

Put a drop or two in water: notice that it does not mix and is oily. Now add two or three drops of concentrated nitric acid to the contents of the dish, and note the change of color. Then add a few drops of water and ammonium thiocyanate: red color indicates iron.

Vitellin.—Make a solution in a small amount of 10 per cent. salt solution; filter and test as follows:

- 1. Add to larger bulk of water made faintly acid with acetic acid.
  - 2. Heat to 75° C.
  - 3. Nitric acid test.
- Shell.—I. Examine a portion of the shell under the low power of a microscope; note the physical character. Treat a portion of the shell with dilute silica of soda solution: when dry examine as before. (Silicate of soda is used for preserving eggs).
- 2. Crush and grind the shell, thoroughly extract with warm water, dissolve the extracted mass with dilute hydrochloric acid. Note the effervescence. Hold in the fumes a drop of lime-water on the end of a glass rod and note the clouding. What gas is formed? Filter the HCl solution and make slightly alkaline with ammonia, add ammonium oxalate and note the white precipitate of calcium oxalate, insoluble in acetic acid. From the data

found give the composition of the shell and the changes which have taken place.

- 3. Allow an egg to stand in strong vinegar for several hours, remove, wash in one change of water, and note the peculiar condition of the egg. Examine the acid liquid as in the preceding experiment.
- 4. Examine equal portions of the yolk and the white of egg, for sulphur by mixing with lime and testing with the lead acetate method given under proteins. Which do you think contains the greater amount of sulphur?
- 5. Weigh an egg accurately and repeat the weighing for five or six succeeding days. Record the results and explain.

## GELATINE

By prolonged boiling with water gelatine is produced from collagen which is a protein occurring in the connective tissue.

Place small pieces of gelatine in contact with cold water and note any change which takes place, then slowly heat the mixture to boiling: does it coagulate? Cool a portion of the liquid: what happens? Test the remainder of the warm solution, divided into seven parts with:

- 1. Dilute hydrochloric acid.
- 2. Acetic acid or lemon juice.
- 3. Picric acid.

- 4. Basic acetate of lead.
- 5. Salt and tannin.
- 6. Heller's test.
- 7. Biuret test.

Note. Any response to Millon's test shows albumen or globulin (impurities).

#### COMPOSITION OF BONES

Bony tissue consists of a mixture of mineral matter (largely phosphate of lime) with protein (ossein). The presence of both is easily shown by simple processes. If fresh bones be steeped for several days in 10 per cent. hydrochloric acid, the mineral matter will be removed and a shrunken but flexible cast will remain. On the other hand, the mineral matter of bone will persist after long careful heating in its original shape. The same result is accomplished much more slowly by weathering.

Procure raw shin bones of beef and have them well scraped and sawed into one inch sections. Treat these sections for two or three hours, under slight pressure, in a soup digester with the least possible amount of water. Pass the extract through cheese-cloth, filter into a tall glass cylinder, and allow it to cool and reserve for future use.

Tests on the extracted bone:

- 1. Dry and examine the bone, comparing its condition before and after treatment.
- 2. Break the extracted bone into small pieces and char a few of these in a small porcelain dish. Note the disagreeable odor of the fumes evolved in the operation. When these have ceased allow the mass to cool somewhat and then transfer to a clean dry test-tube and cork tightly.

Note. This material is known as bone-black and is largely used for decolorizing many food products, notably sugar.

- 3. When the contents of the test-tube are thoroughly cool, pulverize them in a mortar. Test the decolorizing power by placing a portion of the bone-black in a dry filter-paper and passing diluted molasses through it, repeating the operation, if necessary, and compare with the unfiltered portion.
- 4. Ignite another portion of the bone-black on a platinum foil until a white ash is obtained. Dissolve this ash in dilute nitric acid and test for lime and phosphoric acid in the usual manner.

Tests on the Water Extract:

- 1. When thoroughly cool, remove the layer of fat and compare with tallow obtained in a previous experiment.
- 2. Make tests for protein (biuret) and gelatine on the balance of the extract.

#### MUSCLE

The muscle mass consists of a series of elongated tubular sacks of yellow connective tissue (elastic) more or less filled with a mixture of the proteins myosinogen and haemoglobin, mineral matter and water. The tubes are arranged in bundles held together by white connective tissue (collagen), interspersed in the mass are fat globules.

While living muscle contains myosinogen at death it is clotted by enzyme action, the globulin myosin resulting. This in turn is slowly softened by the acids set free by bacterial action (putrefaction) during "hanging."

The muscle tubes are not affected by ordinary cooking processes, while the white connective tissue gradually yields gelatine even at temperatures below boiling. Fresh muscle usually contains glycogen but on standing this is rapidly replaced by lactic acid.

Experiments on Muscle.—Cut off the exterior of a piece of lean meat, test the interior with litmus paper and note the reaction. Then cut the meat in small pieces, pass through a meat chopper and grind the resulting mass in a mortar with clean, dry sand. Take one-half of the ground mass and extract in a beaker of cold water, stirring every few minutes. Allow the extraction to proceed for half an hour; while this is proceeding, take

the balance of the ground mass and extract with 10 per cent. salt solution, stirring as before. Finally pour off and filter the watery extract; divide into four parts, testing each as follows:

- 1. Biuret.
- 2. Heat.
- 3. Add crystals of ammonium sulphate to saturation.
- 4. Boil with a few drops of hydrochloric acid, neutralize with caustic potash, add Fehling's solution and boil: note the result (glycogen).

Saline solution must stand at least one hour (better twenty-four); after standing pour off the solution of proteins and test as follows:

- 1. Pour a few drops into a large excess of water, milky deposit of myosin, soluble in 10 per cent. salt solution.
- 2. Heat short of boiling and note the result: cool, and test the liquid with litmus paper.
- 3. Saturate with salt, precipitate of myosin, filter, dissolve precipitate in weak salt solution (10 per cent.), and make biuret test.
- 4. Suspend a crystal of rock salt in the solution and note the result.

Make a water solution of meat (without washing out the blood), heat to coagulate the protein and filter. To the filtrate, add a few drops of concentrated HNO<sub>3</sub>; evaporate the liquid to dryness and ignite. Cool and take up with water; if cloudy, filter. Divide into five parts and test as follows:

- 1. For chlorides with nitric acid and silver nitrate.
- 2. For sulphates with hydrochloric acid and barium chloride.
- 3. For phosphates with nitric acid and ammonium molybdate.
- 4. For calcium with ammonium hydroxide and ammonium oxalate.
- 5. For iron with hydrochloric acid and ammonium thiocyanate.

Test on Homemade and Commercial Extracts:

Make meat extract by steeping lean meat in cold salt water, gradually heating to a boil and finally under slight pressure. Pour off the liquid, cool, remove the fat, dissolve some of the jelly in warm water and compare with Liebig's and other meat extracts made on the commercial scale, by the following tests:

- 1. Biuret.
- 2. Glycogen test (Iodine).
- 3. Creatinin, Weyl's test: add a very dilute freshly prepared solution of sodium nitroprusside and cautiously caustic soda—ruby-red changing to straw color.
  - 4. Examine the solid extract under the microscope and

note the cubical crystals of salt and knife-rest forms of creatin.

5. Clarify beef extract with white of egg, filter and test filtrate for protein with Biuret test. Compare with test on beef extract before clarifying.

## Chapter XIII

#### MILK

This term usually expresses cow's milk in market condition. Experience has shown that this is a liquid consisting of 87 per cent. of water and 13 per cent. of solids which 3 per cent. is fat and in consequence this composition is now required by law. The three per cent. of fat, technically known as butter-fat, is not in solution but in suspension and separates almost entirely mixed with more or less of the soluble solids, on standing and is called cream. The balance of the solution is known as skim milk.

The following gives a fair idea of the more important ingredients and their quantities:

Water	87.00
Fats	3.00
Protein	4.30
Sugar	5.00
Salts	0.70
-	

The fats in suspension are in the form of minute globules (emulsion) coated with the soluble constituents and will not run together in mass until the coatings are removed by the action of acids (lactic) produced by

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bacterial development some heat and mechanical agitation (churning). The protein matter consists largely of caseinogen, with smaller amounts of albumen and globulin. All milk sours, *i.e.*, becomes acid due to formation of lactic acid at the expense of some of its lactose.

$$C_{12}H_{22}O_{11}$$
,  $H_2O = 4C_3H_6O_3$ .

When the amount of lactic acid reaches approximately 0.5 per cent., the caseinogen precipitates without change of composition (curdles). On neutralizing with alkali, it goes back into solution. As milk is a saturated solution of its constituents, any removal of water usually by means of heat, produces a precipitate which makes its appearance on the surface in the form of a skin. Break this and another forms and so in like manner until no more liquid is left. No coagulation takes place except in the case of the very small amount (0.5 per cent.) of albumen present.

The mineral matter consists very largely of phosphates of lime, whose peculiar function in keeping the caseinogen in solution is well known. Soluble citrates of lime and magnesium are also present in considerable quantities, together with the chlorides of potassium and sodium.

Fresh milk reacts acid and alkaline to delicate litmus paper, due to presence of acid and alkaline reacting phosphates. Old milk is always acid. Specific gravity of milk varies from 1.028-1.033.

#### TESTS

Physical.—I. Cream Gauge.—Fill to mark with freshly mixed milk. Allow the tube and contents to rest quietly for half an hour and read off percentage of top milk from graduated scale.

- 2. Lactometer.—Fill a tall jar with freshly mixed milk, temperature 60°-70° F. Immerse the instrument and when it comes to rest read off the percentage of purity on the scale. In similar manner, determine the purity of skim milk. Finally, add water and redetermine the purity; how can you explain the result.
- 3. Pioscope Test.—Depends on opacity. Place a drop or two of freshly mixed milk in the center of the hard rubber disc. Cover carefully with the glass plate and compare with the standard scale of colors.
- 4. Lactoscope Test.—Use Feser's lactoscope. Fill the pipette with milk, allow it to run into the cylinder. Cautiously add water, shaking after each addition, until the marks on the cloudy glass rod are just visible through the liquid, read off and record the percentage of fat at the level of the liquid.
- 5. Microscope Test.—Examine a drop of milk under the microscope; add a drop of 10 per cent. caustic soda and re-examine. What is the result?

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Chemical Tests.—I. Using fresh milk, what is the reaction with delicate litmus paper?

2. Babcock Test (Determination of Fat).—This test depends on the decomposition of the organic constituents, with the exception of the fats, which are at the same time set free in the liquid state and may be measured.

Fill the milk pipette (17.6 cc.) with freshly mixed milk discharging the contents into the Babcock bottle, add an equal volume of oil of vitriol (sp. gr. 1.8). Mix by revolving the bottle gently in a small arc, back and forth, until the residue disappears and the mass is brown in color. Make tests up in duplicate and whirl them for 5 minutes in the centrifuge over hot water. Stop the machine, add enough warm water to bring liquid level half way up the graduated neck of bottle. Replace them in centrifuge and whirl three minutes, allowing machine to run down. Take out bottle and read per cent. of clear yellow fat floating on the water.

3. Separation and Identification of Caseinogen.—Dilute some milk with 10 volumes of water and carefully neutralize with dilute acetic or hydrochloric acid; no precipitate appears: why? Cautiously add more acid until there is a copious precipitate (caseinogen). This action is hastened by heating to 70° C. Filter through a moist fluted paper and wash well. Reserve the clear filtrate for test B.

- A. Residue of Caseinogen.—Treat residue on the filter paper with a moderate amount of warm 10 per cent. salt solution, pouring it through the filter several times. Add some of this liquid carefully to a saturated solution of salt, adding dry salt if necessary. Observe the precipitate (caseinogen) which dissolves on addition of water.
- B. Divide the filtrate (reserved) into three equal positions.
- 1. Heat to 70°-75° C. and observe the precipitate (lactalbumen). Filter, test precipitate by biuret and filtrate for lactose with Fehling's reagent.
- 2. Add potassium ferrocyanide and excess of acetic acid. Observe the precipitate of lactalbumen,
- 3. Heat to 70°-75° C., filter off lactalbumen, boil the filtrate and observe the precipitate of insoluble calcium citrate. Reserve filtrate.

Note.—If milk has been thoroughly pasteurized, it will not respond to these tests.

C. Evaporate the filtrate, from last test, to dryness; ignite in the presence of a few drops of HNO<sub>3</sub>, cool dilute with water and test for chlorides, sulphates and phosphates.

Quantitative Analysis of Milk.—Measure 5 cc. of milk with a pipette, transfer it to a weighed shallow porcelain dish and weigh again. Difference is weight of milk. Place over hot water (kept just below the boiling

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point) to evaporate water present in milk. Cool and weigh; loss is water, residue is total solids. Total solids should be 12-13 per cent. To extract fat, add about 10 cc. of ether to contents of dish, heat over warm water one or two minutes, decant solution into a second weighed dish. Repeat the ether treatment three times. When dry, weigh original dish; the loss is fat. Evaporate ether from second dish, weigh; the gain is fat and should check the loss.

To extract lactose and soluble salts, treat contents of dish with warm water. Allow it to stand for several minutes, decant the liquid; repeat the operation three times. Dry the dish and weigh; loss is lactose and half the mineral salts found in milk.

**Protein and Insoluble Salts.**—Ignite the contents of dish to a gray ash; protein matter will burn off. Cool and weigh; the loss is protein, residue is insoluble salts. Assuming that insoluble salts are one-half of the total salts, double the figure obtained. To determine amount of lactose, subtract one-half of total salts from the figure obtained on lactose and soluble salts. The difference is the amount of lactose.

Determination of Lactose Quantitatively.—Into a glass stoppered cylinder, put 100 cc. milk and 2 cc. Millon's reagent. Mix thoroughly and pour into a beaker placed over hot water. Allow the mixture to stand until all

protein matter has precipitated, filter off the clear whey through moist fluted paper. Make it alkaline with dry sodium carbonate, adding a little at a time until pink litmus paper turns blue. If cloudy filter again. Pour into a burette and deal with it as with sugar. Calculate that 68 mgs. will reduce 10 cc. Fehling's reagent.

Effect of Rennet.—1. Heat one cup of milk to the boiling-point, boil gently for five minutes, cool to 40° C. and add rennet; note the character and amount of clot.

- 2. Boil one cup of milk 15-20 minutes, replacing any liquid lost during evaporation by hot distilled water; cool to 40° C., add rennet, note character and amount of clot.
- 3. To one cup of milk, add 1-2 cc. of ammonium oxalate solution (precipitant for lime) boil for 2-3 minutes, cool to 40° C. and add rennet; note character and amount of clot, if any. Finally add lime-water equal in bulk to the original milk, warm to 40° and note the result.
- 4. Note the effect of rennet on separate portions of milk heated to 30°, 40°, 50°, 80° C. Tabulate the results of the above tests.

Butter-Fats.—Half fill two 6 in. test-tubes, one with whole milk and the other with skim milk. Add to each half a volume of ether and a few drops of caustic soda, cork and shake well. Uncork and place in a beaker of warm water

and allow it to remain quiet. In a few minutes, note the layer of oil and ether floating on the surface. Remove some of the ether layer from each with a pipette and evaporate at a low heat. Note the difference in amount of the butter residue.

Souring.—Place some milk in a wide-mouthed bottle, allow it to stand in a warm place for some days or until sour. Finally filter off the curd and test the filtrate for lactose and for acidity by titrating with 10/N alkali, calculating to lactic acid. What weight of bi-carbonate of soda would neutralize the amount of acid found? Condensed or evaporated milks should be diluted with distilled water to the original bulk and treated as normal milks.

Preserved milks commonly contain cane-sugar. Dilute a sample to the original bulk, precipitate the caseinogen with dilute acetic acid; filter and exactly neutralize the filtrate with sodium carbonate and test for sucrose with cobalt chloride and caustic soda.

The presence of formalin in milk will be noticed in the Babcock test, by the appearance of a violet band at the junction of the acid and oily layers.

Borax and borates will be found with the ash.

## EXPERIMENTS ON CHEESE

Take a sample of any well-cured cheese, grind some of it in warm water, filter and reserve the residue. Divide the filtrate into six parts and test as follows:

- 1. For acidity or alkalinity with litmus paper and N/10 acid or alkali.
  - 2. For soluble protein.
  - 3. Neutralize for meta-protein.
  - 4. For peptone.
- 5. For soluble mineral matter, *i.e.*, chlorides, sulphates, etc.
  - 6. For ammonia and sulphides.

Extract the residue several times with the same portion of warm alcohol and test the extract for fatty acids. Again extract the residue with warm ether several times and evaporate some of the clear ethereal liquid over warm water. Is the residue greasy?

Divide the extracted residue into two parts and test as follows:

- 1. For insoluble protein.
- 2. Burn to white ash and test for insoluble mineral matter,—phosphates, lime, etc.

During the incineration, hold pieces of moistened red litmus and lead acetate papers in the fumes and record the results.

Cheeses are frequently preserved in wrappings saturated with borax or boracic acid solution. To determine this, steep some of the paper wrapping in warm water, filter if necessary, acidify with HCl and dip pieces of

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turmeric paper in the liquid. Dry these at 212° F., a pink color indicates borates.

## CHEESE

A product prepared from the caseinogen of milk with or without the fat. The milk is clotted with rennet, separated from the whey, ground, salted, pressed into shape and cured. The curing operation consists in subjecting the cheese mass to the action of certain bacteria and moulds, which form acids, hydrolyze the proteins and develop flavor and odor.

Cottage cheese is merely finely divided caseinogen precipitated by the lactic acid of the souring process and undergoes no further change.

Cheeses are usually made from cow's milk but may be produced from goat's or ewe's milk or mixtures of all of them.

# Chapter XIV

### FERMENTS AND PRESERVATIVES

The organisms which cause the most common changes in our food materials are generally known as yeasts, lactic acid and vinegar ferments. Their spores are present in all house dust. These organisms are distinguished by the fact that they operate in presence of air, under widely varying temperature conditions, and give off no disagreeable odors while their products are nonpeisonous. It is true that putrefactive bacteria play some part in the preparation of our food, notably in meats and cheeses, but great care must be observed that the process is kept under strict control and only allowed to proceed to a limited extent. Yeasts so familiar as producers of fermentation, work on starch, maltose, sugar and the glucoses. At least two enzymes are secreted by these organisms, i.e., invertase, changing starch, maltose and sugar into glucose; and zymase, changing the glucose into carbon dioxide and alcohol. These changes occur between 32° and 122° F., and are at a maximum at 92° F. When 15 per cent. of alcohol has been formed even if the material contains unchanged carbohydrate, the action ceases. Yeasts are not easily destroyed by cold unless exposed to very low temperatures for long

periods. Above 122° F., however, they are quickly killed so that it is not necessary to reach the boiling point 212° F. (sterilization). If a lower temperature be employed, it is well to maintain it for some time to insure uniform heating of the mass (pasteurization). Varieties of yeasts are known as Brewer's, Distiller's and Wild.

Lactic acid bacteria chiefly attack the sugar of milk, working until about one per cent. of acid is formed, but always leaving much of the carbohydrate unchanged. They are not easily destroyed by cold but do not act below 50° F. and continue their work up to 130° F., being most active at 110° F. Conditions for sterilization and pasteurization are similar to yeasts. The other carbohydrates are less susceptible to this ferment; sour bread and sauerkraut, however, are the result of their work.

Acetic or vinegar ferments act on all weak alcoholic liquids; 10 per cent. and under. The temperature conditions are much the same as for lactic acid (50°-110° F.). Fermentation ceases when 5 per cent. of acid has been produced. The ferment is commonly known as "Mother of Vinegar." Conditions for sterilization and pasteurization are similar to yeasts.

Yeast—Temperature Experiments.—Prepare four sixinch test-tubes with perforated corks, bearing tubes bent in the form of an inverted letter J. Fill three of the tubes with a mixture, prepared from one-half a yeast

cake, one-half tablespoonful of molasses and a cup of water. Fill the fourth with the same preparation filtered through absorbent cotton. Allow tubes Nos. I and 4 to stand, while No. 2 is subjected to a temperature of 32° F. (produced by a mixture of pulverized ice and salt) for fifteen minutes. No. 3 is boiled for two or three minutes. Now place the four pieces of apparatus so that the delivery tube of each reaches to the bottom of a test-tube containing about two inches of clear limewater, and allow them to stand for at least twelve hours in a warm place (90° F.). At the end of this time, examine each tube of lime-water, first for a precipitate, and second with litmus paper. Finally examine the liquid in the fermentation tubes, noting its odor and general properties.

Action of Yeast on Various Foods.—Prepare two solutions of sugar in water as follows: For the first use equal quantities of granulated sugar and water, for the second take one-fourth of the strong solution and dilute with three volumes of water. Fill two of the fermentation tubes already prepared with two sugar solutions (Nos. I and 2). Dissolve one-eighth of a yeast cake in about 30 cc. of milk and pour the mixture into a fermentation tube (No. 3). Fill, a fourth fermentation tube (No. 4) with a mixture of one-eighth of a yeast cake dissolved in thin, clear flour paste. Connect all four of the fermentation tubes with lime-water tubes as before and allow them to stand for twelve hours in a warm

place (90° F.). Examine the contents of each lime-water and fermentation tube.

Acetous Fermentation.—Make a weak solution of alcohol in water (5 parts of alcohol to 20 parts of water) and test with litmus paper; if acid, neutralize with a weak solution of sodium carbonate and test a small portion with potassium iodide and potassium hydroxide, heat—the odor of iodoform shows the presence of alcohol.

Divide the balance of the solution into two equal parts; pour one into a shallow dish and place the other in a well-corked bottle. After the solutions have stood for a week, test with litmus paper, and also by adding alcohol and warming gently. Note the peculiar odor (ethyl acetate—odor of hard cider) in the first case but not in the latter. Explain.

Expose a small quantity of beer to the atmosphere for several days; subsequently examine for acidity with test paper and for acetic acid with alcohol. From the results of these experiments explain why bottled weak alcoholic beverages keep sweet.

Lactic Acid.—To about six ounces of cold pasteurized milk contained in a small flask, add one tablespoonful of the liquid obtained by dissolving one lactobacilline (Metchnikoff) tablet in half a cup of tepid water. Mix well and keep at 100° F. for several hours. Carefully observe all changes taking place and compare with the well known buttermilk.

## FOOD PRESERVATION AND PRESERVATIVES

All foods are subject to the attack of bacteria and in consequence their value is very generally seriously impaired. Methods for prevention of these changes have been used from the earliest times and are known as preservation.

At least two general types of process are in common use, viz., physical and chemical. To the first class belong such methods as drying, cooling and canning. These processes are applicable to all kinds of foods, possess high efficiency and make very slight changes in flavor, appearance and composition. Unfortunately, food materials preserved in any of these ways will change very rapidly with slight variation of physical conditions, hence the effects are not permanent. The second class involve such change of chemical conditions that no matter what physical changes may occur, decomposition cannot take place. The results are permanent but are accomplished at the expense of flavor, appearance, etc.

So general has the use of chemical preservatives become that a brief discussion of the subject seems necessary. The best known and as generally conceded harmless are: alcohol, vinegar, sugar and salt (NaCl). With the exception of vinegar (acids generally being inimical to bacteria) the action seems to depend on making the protein matter present, insoluble; hence we find

the quantity of the preservative important. Well known operations are as follows:

Alcohol—50 per cent. "Brandying."
Salt—dry or supersaturated solution "Pickle."
Sugar—syrup—solutions of 25 per cent. or more.

Less well known methods accomplish similar results by using very small, in some cases minute proportions, of other chemical agents; but the actual chemical operation can only be surmised in most cases. Included in this list are: borates, fluorides, sulphites, formaldehyde, benzoates, salicylates and creosote. It may be as well to observe that the use of spices, for instance in mince meat, is c rtainly parallel with benzoates and salicylates.

#### EXPERIMENTS

Alcohol and vinegar are first separated by distillation and then identified by well known methods. Sugar and salt may also be determined by diluting, filtering and testing the clear filtrate.

Borates.—Ash some of the substance, cool, make strong water extract, filter if necessary and neutralize with dilute HCl. Dip a strip of turmeric paper in this liquid, remove and dry by steam heat. (This may be accomplished by wrapping the moist paper around the upper part of a test-tube partly filled with water and boiling gently). The paper turns pink on the edges.

Or moisten the ash in the dish with alcohol, add 8 to 10 drops of glycerine, mix well with a glass rod and ignite the mass with a match or Bunsen burner. Note the yellow flame with a green edge, characteristic of borates.

Fluorides.—Mix the liquid or solid mass with an excess of lime-water, evaporate to dryness, ignite, cool and make the etching test.

Sulphites.—If present in quantity, they are distinguished by their odor and taste "sulphur match" especially on warming.

For small amounts of sulphites mix with bromine water, boil off excess and test for sulphates.

Formaldehyde.—Conc. H<sub>2</sub>SO<sub>4</sub>, in the presence of ferric salt, produces a violet color. (Oil of vitriol contains enough ferric salt to act as a satisfactory reagent).

Benzoates.—Carefully mix liquid substance with onetenth of its volume of chloroform and a few drops of oil of vitriol. Avoid violent shaking (mix with a circular motion). Allow the mixture to remain quiet until chloroform layer separates out. Remove some of this layer with a pipette and evaporate it in a clean porcelain dish over hot H<sub>2</sub>O. Note the flat crystalline plates of benzoic acid, which give off a pungent odor on heating. Examine the original mixture in the flask; note any violet color between layers of acid liquid and chloroform. This indicates salicylic acid.

Both benzoic and salicylic acids are not present in the same liquid.

## Chapter XV

#### BAKING POWDERS

It is frequently necessary to develop carbon dioxide for leavening purposes more rapidly than by the agency of yeast. For this purpose the purely chemical method by the acid decomposition of carbonates or bicarbonates is most available.

Undoubtedly the time-honored custom of using saleratus (bi-carbonate of potash) and sour milk (lactic acid) furnished the original ideas on which the modern mixtures were built up.

This original idea still survives to some extent in modern practice, but is open to at least two strong objections. First that bi-carbonate of potash is no longer a commercial article but is replaced by the cheaper and stronger bi-carbonate of soda; still no change is made in the proportions used. The quantity should be reduced nearly one-sixth.

Second that it is very difficult to estimate the amount of lactic acid in sour milk by simple means with any accuracy. In fact the quantity is usually largely overestimated. When milk shows decided indications of the sour stage only four-tenths of one per cent. of lactic acid are usually found.

It must be remembered that any excess of the bi-carbonate used, is changed into alkaline normal carbonate by the heat of baking.

For the above stated reasons it can easily be seen that accurately compounded mixtures (leaving neither alkaline or acid residues) and retaining their qualities for some time in the dry state, but ready to develop gas on addition of water have a decided advantage.

In order to preserve these mixtures in a dry state, it has been found advisable to add to them such agents as raw starch and pulverized lactose which are perfectly harmless, and dry alum which is deleterious; such additions should not in any case exceed 25 per cent. of the whole mass. When used for this purpose the compounds are known as "fillers."

Modern baking powders may be classed as tartrate, phosphate, and alum phosphate. All contain bicarbonate of soda, while the acting acid ingredient varies, as follows:

Tartrate—Cream of tartar,  $KHC_4H_4O_6$ , and sometimes a small amount of free tartaric acid,  $H_2C_4H_4O_6$ .

Phosphate—Soluble phosphate of lime,  $CaH_4(PO_4)_2$ . Alum phosphate, same as above with addition of dry alum  $K_2$ ,  $Na_2$ , or  $(NH_4)_2$ ,  $Al_2(SO_4)_4$ .

The following reactions clearly show the changes taking place in using these mixtures.

For tartrates:

$$KHC_4H_4O_6+NaHCO_3=KNaC_4H_4O_6+CO_2+H_2O.$$
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For phosphates:

$$CaH_{4}(PO_{4})_{2}+2NaHCO_{8}=CaHPO_{4}+$$
 $^{136}$ 
 $Na_{2}HPO_{4}+2CO_{2}+2H_{2}O.$ 
 $^{142}$ 
 $^{88}$ 
 $^{36}$ 

For alum phosphates:

$$(NH_{4})_{2}Al_{2}(SO_{4})_{4}+CaH_{4}(PO_{4})_{2}+4NaHCO_{3}=$$

$$Al_{2}(PO_{4})_{2}+CaSO_{4}+(NH_{4})_{2}SO_{4}+$$

$$^{245}$$

$$^{136}$$

$$^{132}$$

$$^{2}Na_{2}SO_{4}+4CO_{2}+4H_{2}O.$$

$$^{284}$$

$$^{176}$$

$$^{72}$$

It is significant that the tartrate powders leave no insoluble residue except starch, while the others leave nearly one-third of their weight in insoluble mineral matter besides the starch. The phosphates yielding the acid soluble phosphate of lime of doubtful utility, and the alum powders, alumina phosphate and calcium sulphate which are positively detrimental.

#### **EXPERIMENTS**

Tartrates.—Mixtures of cream of tartar and bicarbonate of soda with starch or lactose filler. Treat a small portion of the powder with water and after the effervescence has ceased test a portion of the liquid for starch with iodine solution and for lactose with Fehling's solution, boil the remainder of the liquid, cool, filter through fluted paper, and test with litmus paper.

- 1. Place a few drops of the clear liquid on a slide and allow it to evaporate spontaneously. Examine the cleft rectangular crystals of Rochelle salt.
- 2. Test another portion of the solution by adding one drop of fresh cold solution of ferrous sulphate, one or two drops of peroxide of hydrogen and a large excess of caustic potash—a violet color due to tartrates. Evaporate the balance of the solution in a porcelain dish, char and gently ignite the residue. Note the odor while carbonizing, what does it suggest? Cool, add water and test with litmus paper: why is it alkaline?

Neutral tartrates will yield to the silver mirror test.

Tartrate powders may contain a small amount of bicarbonate of ammonia. To test for this, heat a portion of the powder in a test-tube with caustic potash solution; observe the odor; or hold a strip of moistened red litmus paper over the mouth of the tube.

**Phosphate Powders.**—Calcium hydrogen phosphate, bicarbonate of soda and starch filler. Make a water solution and test for filler as in the case of phosphates. Divide the remainder of the solution into three parts.

1. Make acid with nitric acid, add a few cc. to ammonium molybdate and warm—yellow precipitate indicates phosphates.

- 2. Add ammonium oxalate, and ammonium hydroxide until alkaline and boil—white precipitate indicates calcium. This should give a yellowish red flame on platinum wire.
- 3. As these powders frequently contain alum it is necessary to make a test. A portion of the solution placed on a slide and allowed to evaporate spontaneously will yield large truncated octahedra of alum. Probably the best method for the determination of alum is to add a portion of the solution to tincture of logwood diluted with two or three volumes of water, finally adding an equal volume of ammonium carbonate. In the presence of alum the liquid is colored lavender or dark blue.

Carbon Dioxide Determination by the Scheibler Apparatus.—Weigh out 500 mgs. of the baking powder place in the glass-stoppered bottle belonging to the apparatus. Put a small quantity of water in the gutta percha tube (two-thirds full). The columns of water in the apparatus will be at the same level when the pressure inside of the apparatus is the same as the atmospheric pressure, and this should be the condition when the experiment is started. The gutta percha tube is placed inside the bottle containing the 500 mgs. of baking powder and the apparatus is then connected up. Be sure the relief valve is open when the apparatus is put together and closed immediately afterwards. Incline the generat-

ing bottle to allow the water to come in contact with the powder. Observe the evolution of gas. Note the height of the water column. Shake the generating bottle vigorously until no more gas is evolved. Immediately afterwards balance the water columns by allowing some water to escape into the overflow flask. Read the figure nearest the level of the water. This reading indicates the per cent. of gas liberated by the addition of water to the baking powder, or in other words, the leavening power of the baking powder. This reading should be in the neighborhood of 10 per cent. in a fresh tartrate powder.



## Chapter XVI

## TEA, COFFEE, CHOCOLATE AND COCOA

Tea consists of the cured, dried and rolled leaves of a variety of plants known as the Thea. According to the age of the leaf gathered, there are four well known grades, Pekoe the youngest, Souchong next, Congou next and Bohea the oldest. All these are found in the grades of green or black as the method of curing varies. Green teas are not fermented, black teas are fermented, and since fermentation tends to reduce the amount of tannin. the latter are very generally preferred. The infusion is made by steeping the leaves in freshly boiled water (preferably slightly hard) just below boiling. Five minutes is sufficient to make the extract, when it will contain the maximum of oil, extract and caffein and the minimum of tannin. It should now be poured off the leaves and used. Boiling or long standing increases the amount of tannin in the infusion, while it does not materially affect the caffein or extract.

Experiments on Tea.—Make an infusion according to rule given in the text, pour off the clear liquid, filtering if necessary, and examine the leaves with a magnifier. Add a few drops of the clear filtrate to a weak starch solution faintly colored with iodine; if tannin is present

the color will fade. Mix the balance of the tea extract with 1/10 of its volume of chloroform and when settled draw off the lower layer and evaporate in a porcelain dish over hot water, the residue should be almost colorless, crystalline and very bitter (Caffein).

Evaporate some of the tea extract after removing the chloroform in a clean porcelain dish over hot water and note the large amount of residue, also its color and gummy nature.

Coffee consists of the dried fermented and roasted beans of the caffoea arabica. The extract is made from the finely pulverized beans, which are first mixed with cold fresh water gradually brought to the boiling point, and held there two or three minutes. After settling of the insoluble residue, the clear brown liquid is ready for use. Settling can be hastened by the cautious addition of cold water or a coagulating agent (white of egg).

Coffee extract contains about the same amount of caffein and tannin as is found in tea extract but larger quantities of gum, dextrine, etc.

French coffee usually contains chicory, the kiln dried root of the wild endive; the drying operation produces caramel at the expense of sugar and hence the water extract is darker in color.

Coffee substitutes are composed of roasted cereals or breads with or without the addition of ground roasted coffee. Their extracts may be entirely free from caffein and tannin, but in any case will contain less than genuine coffee. The bitter taste and dark color is due to caramel.

**Experiments on Coffee.**—Grind the roasted beans to a fine powder, throw half a teaspoonful of the powder into a vessel holding cool water, stir well, and note whether any color is imparted to the liquid (chicory).

Moisten one tablespoonful of the powder with cold water, add one cup of warm water, bring to the boiling point and boil two minutes. Filter through paper or cotton and reserve the clear filtrate for test as follows:

Decolorize a small portion with bone-black and when cold test for starch with iodine. It should be absent; if present the sample contains cereal or bread.

Test another portion for tannin (see tea). Determine presence of caffein (as under tea). Chill some of the clear filtrate; should it turn cloudy, make further test for dextrine with alcohol.

Examine thoroughly extracted coffee grounds under the microscope.

Determine quality of ash.

Chocolate and Cocoa.—These products are made from the fermented and dried seeds of the fruit of the theobromo cacao, which resembles the cucumber. After drying and husking, the seeds yield two halves called "nibs."

The nibs are ground to a fine powder under hot rolls

which melts the large quantity of fat (cocoa butter) present and produces a liquid mass. If this is allowed to run into shallow molds and cooled the product is called chocolate or bitter chocolate. Sugar and vanilla extract are often added to the liquid before cooling, producing sweet or edible chocolate.

If the fluid mass of ground nibs is pressed to remove fat and the remainder is cast in molds and afterward ground, the product is called soluble cocoa. Alkali in small amount is frequently used to make the cocoa more soluble.

Cheap grades of cocoa contain considerable quantities of starch and ground cocoa shells.

Experiments on Chocolate and Cocoa.—Boil some of the finely ground mass with water, filter while hot and reserve both filtrate and residue for test.

Tests on Filtrate.—For starch, dextrine, sugar, protein matter and soaps.

Tests on Residue.—Dry and extract fatty matter with gasolene. Examine extracted residue under the microscope for fiber. Determine quality and amount of ash.

## Chapter XVII

#### STAINS

Removal of stains depends on the nature of the fabric, quality of the dye and character of stain.

### I. Fabrics:

- 1. Silk-most easily damaged.
- 2. Wool-next.
- 3. Cotton-next.
- 4. Linen-least.

## II. Dyes:

- 1. Natural dyes least liable to injury.
- 2. Artificial dyes (except indigo and alizarine) most susceptible to change.

## III. Solvents:

- 1. Water.
- 2. Alcohol.
- 3. Ether.
- 4. Gasolene.
- 5. Chloroform.

### IV. Absorbents:

- 1. Talc.
- 2. Starch.
- 3. Paper.

## V. Detergents:

I. Neutral soda or potash soaps.

#### VI. Bleaches:

- 1. Peroxide of hydrogen.
- 2. Hypochlorites of sodium or potassium.
- 3. Hyposulphite of sodium.
- 4. Sulphites and SO<sub>2</sub>.

## VII. Neutralizing Agents:

- 1. Ammonia.
- 2. Oxalic acid or acid oxalate of potassium.
- 3. Muriatic acid, very dilute (HCl).
- 4. Acetic acid.

Steaming, used when softening old stains or in very delicate fabrics.

## VIII. Removal of stains and spots, caused by:

- 1. Fatty bodies as grease or oil.
- Use (a) Soap and water.
  - (b) Ammonia and water.
  - (c) Talc.
  - (d) Dry starch.
  - (c) Paper.
  - (f) Ether.
  - (g) Gasolene.
- 2. Fruits.

Coloring matter.

(a) Hot water.

(b) Bleaches—salts of lemon (binoxalate of potash). Acids.

Use dilute ammonia.

Note:—Do not put ether or gasolene on a wet fabric. As gasolene and other fatty solvents tend to spread grease spots, it is well to mix them with talc or starch and apply to the spot. When dry, brush; repeat if necessary.

- 3. Mineral matter.
- (a) Rust—
- Use (1) Mineral acids, HCl, (on dyed fabrics).
  - (2) Organic acids (oxalic, citric or tartaric).
- (b) Acids-

Use ammonia.

4. Fungoid growths as mildew.

Use milk of lime and a bleach.

- (a). Javelle water.
- (b). Labarraques solution.
- (c). Ammonia and peroxide of hydrogen.
- (d). Sunlight.
- (e). Milk.
- 5. Ink-Iron base-
- Use (1) Ordinary ink if not too dry, yields to warm alcohol.
  - (2) Salts of lemon.
  - (3) Oxalic acid.

6. Sugar and gum—

Use warm water only.

7. Paint and varnish-

If moist—use gasolene.

If dry—soften with amyl acetate or pine tar oil and then remove with gasolene.

8. Scorch-sunlight.

9. Bluing-boil with dilute acid or alkali until removed.

## Chapter XVIII

## REAGENTS

Commercial		Laboratory strength						
forms	Sp. gr.	Per cent.	Concentrated	Sp. gr	Dil	utc	Sp. gr.	
Acids					Vols. H <sub>2</sub> O	Vols.		
HC1	· I.2	40	full strength	1.2	I	I	I.I	
			Vols. Vols. $H_2O$ acid					
$HNO_3 \cdots$	· 1.4	70	I I	1.2	3	I	1.1	
$H_2SO_4 \cdots$	. 1.84	94	full strength	1.84	7	1	I.I	
CH₃COOH ···	. 1.06	50	full strength	1.06	10	I	1.007	
Alkalies					Vols. H <sub>2</sub> O	Vols. alk.		
NH <sub>4</sub> OH	• 0.9	28	full strength		I	I	0.945	
NaOH			20 per cent.	1.3	10 ре	r cent	. 1.14	
кон	•		20 per cent.	1.23	10 ре	r cent	. 1.1	
Salts								
$Na_2CO_3 \cdot \cdot \cdot \cdot$	•		Dry		10 pe	r cent	1.1	
BaCl <sub>2</sub>	• •				10 ре	r cent		
$(NH_4)_2C_2O_4\cdots$					satur	ated		
Na <sub>2</sub> HPO <sub>4</sub> ·····	• •				satur	ated		
NH,C1					10 ре	r cent		
(NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> ···	•		Dry		satuı	ated		
NaCl	•		Dry		satur	ated	1.2	
$MgSO_4 \cdots$			Dry		satur	ated		
HgCl <sub>2</sub>					satur	ated		
Tannin					20 pe	r cent		
$AgNO_3 \cdot \cdot \cdot \cdot$					5 pe	r cent		
$Co(NO_3)_2 \cdot \cdot \cdot \cdot$					10 ре	r cent		
K, Fe(CN),					10 ре	r cent		

#### SPECIAL REAGENTS

Ammonium Molybdate, (NH<sub>4</sub>)<sub>2</sub>MoO<sub>4</sub>.

Dissolve 100 grams MoO<sub>3</sub> in 400 cc. strong NH<sub>4</sub>OH and slowly pour resulting solution in 1500 cc. HNO<sub>3</sub> sp. gr. 1.2.

Magnesia Mixture.

1 gram MgSO<sub>4</sub>, 1 gram NH<sub>4</sub>Cl, 4 cc. ammonia, 8 cc. water.

Millon's Reagent.—100 grams mercury dissolved in 83.3+ cc. HNO<sub>3</sub> sp. gr. 1.2 in the cold, when action ceases add twice the volume of cold water.

Fehling's Reagent.—Solution A—34.64 grams CuSO<sub>4</sub>, 5H<sub>2</sub>O in 400 cc. of cold water, when dissolved make up to 500 cc.

Solution B—50 grams NaOH+180 grams NaKC $_4$ H $_4$ O $_6$  in 300 cc. of water, when dissolved and cooled make up to 500 cc.

For use mix equal volumes of A and B and add two volumes of water.

Barfoed's Reagent.—4.0 grams copper acetate, 100 cc. water, 2 cc. acetic acid.

Nylander's Reagent.—Two grams bismuth sub-nitrate, (BiONO<sub>3</sub>), and 4 grams of Rochelle salt, (NaKC<sub>4</sub>H<sub>4</sub>O<sub>6</sub>), in 100 cc. of 8 per cent. NaOH, sp. gr. 1.08.

Nessler's Reagent.—Dissolve 2 grams KI in 5 cc. H<sub>2</sub>O, add 4 grams HgCl<sub>2</sub> solution, or so much that on warm-

ing a little of the ppt. remains undissolved. After cooling, dilute with 20 cc. H<sub>2</sub>O, filter and add 30 cc. of a solution of 1 gram of KOH in 2 cc. H<sub>2</sub>O.

Soap Solution (Stock).—Dissolve 10 grams of good castile soap in 1000 cc. of 90 per cent. alcohol.

For use mix 100 cc. of the above with 100 cc. of distilled H<sub>2</sub>O and 30 cc. of alcohol.

Gries's Reagent for Nitrites.—Dissolve I gram of sulphanilic acid in 300 cc. of acetic acid sp. gr. 1.04 (30 per cent.).

Boil 0.2 gram of  $\alpha$ -naphthylamine in 400 cc. of distilled water, filter through a plug of washed absorbent cotton and add 360 cc. of acetic acid (30 per cent.).

To dilute 50 per cent. acid to 30 per cent., take 3/5 of 100 or 60 cc. of acid and dilute to 100 cc.

Basic Acetate (Sugar of) Lead Solution.—Boil 232 grams of lead acetate and 132 grams of litharge, (PbO), in 750 cc. of distilled water for half an hour, cool, dilute to one liter. Allow liquid to stand until clear and decant. Sp. gr. of solution should be about 1.267.

Alumina Cream for Clarifying Syrups, Etc.—Precipitate a solution of alum with slight excess of ammonia, wash by decantation or filtration until neutral and suspend in water to a creamy consistency.

Neutral Fuchsin.—Dissolve one gram of fuchsin in 100

cc. of distilled water and carefully neutralize with very dilute caustic soda. A few drops will suffice.

This reagent dyes linen pink but fails to affect cotton.

Meta Phosphoric Acid.—Dissolve glacial phosphoric acid, (HPO<sub>3</sub>), or phosphoric anhydride P<sub>2</sub>O<sub>5</sub> in ice and water. As the solution rapidly changes to H<sub>3</sub>PO<sub>4</sub> make it fresh for each day's work.

Alcohol.—95 per cent. is always acid, neutralize with dilute alkali before using. Alcohol may readily be recovered from solutions and wash liquids by distilling over hot water at 78°-80° C.

## List of Apparatus for Students in Household Chemistry

Three rings (iron).

Filter ring.

Clamps.

Triangular file.

Round file.

Triangles.

Wire gauze.

Steel forceps.

Wing top.

Horn spatula.

Tube brushes—three (assorted sizes).

Filter paper.

Test tube holder.

Scissors.

Knife.

Thermometer, Centigrade 0°-120°.

Glass rod with platinum wire.

Flat glasses, 4 inch.

Blue glass.

Watch crystals—four.

Microscope slides with cover glasses—four.

Test-tubes—1 doz. 6-inch.

Test-tubes—1 doz. 4-inch.

Hard glass test-tubes (1 in. x 6 in.)—two.

Graduate, 10 c.c.

Porcelain dishes — two (3½ inch).

Beakers (Jaikel 1-4).

Tripod

Test-tube rack.

Agate boilers with cover, ½ pint.

2 funnels—two inch and three inch.

Flasks-one 4 oz. high.

Flasks—two 4 oz. low (wide mouth).

Flask—one 16 oz. round bottom,

Wash bottle—16 oz.

Wide mouth bottle, 8 oz.

Water bath, 5 in.

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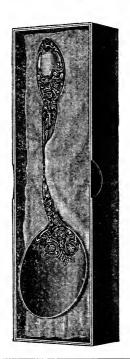
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