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APPLIED CHEMISTRY

[illegible] STUDENTS IN DOMESTIC [illegible]

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LABORATORY WORK
IN
APPLIED CHEMISTRY

FOR STUDENTS IN DOMESTIC SCIENCE

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I. DETERGENTS AND CHEMICALS EMPLOYED IN THE LAUNDRY.

A. WATER FOR LAUNDRY WORK.

Natural Waters Are Solutions.

Principle of the Method.—An accurately measured volume of water to be examined is evaporated in a weighed vessel. The vessels together with the dry solid residues is weighed, the amount of solid residue being determined by subtraction of the two weights.

Apparatus.—Convex cover (10 cms.); 500 c.c. lipped beaker, ring stand, wire gauze, Bunsen burner, 100 c.c. graduated flask, balance and weights.

Details of Procedure.—Place the beaker one-third filled with water on the wire gauze and heat to boiling. While the water in the beaker is being heated, weigh the convex cover to the nearest milligram, first having scrupulously cleaned and dried it. Measure into the graduated flask 100 c.c. of the water to be examined or such a volume as will leave not less than 10 milligrams of solid residue on evaporation. Place the weighed convex cover on top of the beaker containing the boiling water and carefully add a portion of the measured volume of water. As it evaporates, add more and more, until the whole has been evaporated. Continue the evaporation until the residue on the cover appears perfectly dry. Cool in a desiccator and weigh with the same precision as before.

Calculation of Result.—Express the result as parts per million and as grains per U. S. gallon. Consult works in the library or other sources to secure similar data on other waters and arrange them in the form of a table.

Sources of Error.—Aside from mistakes or other inaccuracies in weighing or measuring, serious error may creep in through the accumulation of dust during the evaporation. This may be guarded against by suspending an inverted funnel over the cover.

The Effect of Dilute Solutions of Salts on Soap Solutions.

Principle of the Method.—Soap solution gives suds when shaken with water. The amount of the solution of soap necessary depends upon the proportion of dissolved salts in the water.

Apparatus.—A glass-stoppered bottle (capacity about 200 to 250 c.c.), burette, 100 c.c. pipette.

Reagents.—Alcoholic soap solution, 10 grams powdered Castile soap in 1 liter, dilute alcohol (1/3 water); approximately 0.004 N solutions of aluminum chloride, ferrous chloride, manganous sulphate, calcium chloride, magnesium sulphate.

Details of Procedure.—Measure 100 c.c. of distilled water into the glass bottle. Fill the burette with soap solution. Add the soap solution 1 c.c. at a time to the contents of the bottle, stoppering and shaking after each addition. Continue the addition of the soap solution until a suds is obtained, which persists for five minutes. Record the number of c.c. of soap solution required. Repeat with each of the salt solutions mentioned.

Conclusion.—Record the results in a table.

The Effect of Dilute Solution of Calcium Bicarbonate on Soap Solutions.

Reagents.—Alcoholic soap solution prepared as directed in the preceding experiment; saturated solutions of calcium hydroxide and calcium sulphate.

Details of Procedure.—Pass carbon dioxide gas through 25 c.c. of the saturated calcium hydroxide solution until the precipitate that forms is entirely dissolved. Dilute to 200 c.c. with distilled water. Divide the solution into two equal parts. To the first, add soap solution in exactly the same manner as directed in the preceding experiment. Record the result. Boil the second part until no more CO_2 is evolved. Note the result. (?). Restore the volume to 100 c.c. with distilled water and again test with soap solution. Repeat the experiment, using a solution of calcium sulphate (1 c.c. of the saturated solution diluted to 100 c.c. with distilled water).

Questions.—Write the equations for each step in the above experiment.

What is meant by "Hardness of Water"?

Which part of the above experiment illustrates water of temporary and which water of permanent hardness? Give reasons for your answer.

Determination of the Soap Consuming Power of Water.

Principle of the Method.—A soap solution is standardized by titration against an artificial water of known degree of hardness. Natural waters are examined as to their soap consuming power by titration with this standard soap solution.

Apparatus.—A glass-stoppered bottle of 200 to 250 c.c. capacity, burette, pipettes (10 c.c. and 100 c.c.), 100 c.c. graduated cylinder, graduated liter flask.

Reagents.—Weigh accurately one gram of pure calcite ($CaCO_3$). Dissolve in HCl and evaporate to dryness to expel the excess of acid. Take up with water, add a drop of ammonia and dilute to a liter in the graduated flask. This solution constitutes a standard hard water 1 c.c. = .001 grams $CaCO_3$; soap solution used in the previous experiment.

Details of Procedure.—Place 10 c.c. of the standard hard water, measured by a pipette, in the glass stoppered bottle, add 90 c.c. of distilled water, then soap solution from the burette ½ c.c. at a time, shaking after each addition, until a lather is formed, which persists for five minutes. Record the number of c.c. used. This gives the approximate amount of soap solution equivalent to 10 c.c. of the standard hard water, plus 90 c.c. of distilled. Repeat this operation until three results are obtained, which do not vary in reading more than ½ c.c. This mean reading gives the true value in c.c. of soap solution equivalent to 10 c.c. of the standard water, plus 90 c.c. of distilled. Since distilled water itself requires some soap solution before a lather is obtained, a blank is run in a similar manner to the above on 100 c.c. of distilled water. By subtracting the mean here obtained from the mean number of c.c. obtained in the first instance, the quantity of soap solution equivalent to .01 grams $CaCO_3$ is obtained. Calculate the value of 1 c.c. of the soap solution in terms of $CaCO_3$.

With the solution so standardized, determine the soap consuming power of several waters. Place 100 c.c. of the water to be examined in the glass stoppered bottle and add standard soap solution in exactly the same manner as in the standardization. Read the number of c.c. of soap solution required and calculate the hardness of the water.

Calculation of the Result.—Express the hardness of the water in terms of parts of $CaCO_3$ per million.

Multiply the known value of 1 c.c. of soap solution by the number of c.c. used, and again multiply by 10 in order to express the hardness in parts per million.

Determination of the Temporary Hardness (Alkalinity) of Water.

Principle of the Methd.—If we assume that all the acid consuming substances of the water are calcium and magnesium bi-carbonates, the amount of acid used to neutralize the water is a measure of the temporary hardness.

Apparatus.—1000 c.c. beaker, stirring rod, 100 c.c. pipette, burette, 1000 c.c. flask.

Reagents.—N/10 HCl, methyl-orange (.02 grs. per 100 c.c.).

Details of Procedure.—Measure into the beaker by means of the liter flask 1000 c.c. of the water to be tested. Add not more than 10 drops of methyl-orange and titrate in the usual way with the N/10 HCl until the acid gives the characteristic color of the indicator. Repeat, using 1000 c.c. of distilled water.

Calculation and Statement of Results.—Hardness is to be expressed in parts of $CaCO_3$ per million. One c.c. N/10 HCl neutralizes 5 mgr. $CaCO_3$. (Show how this figure is obtained.) If, therefore, 1000 c.c. of water (1,000,000 mgr.) is used for the titration, each c.c. of N/10 HCl must equal 5 parts of $CaCO_3$ per million.

Note.—The value here obtained is not taken as a measure of the temporary hardness unless the absence of sodium carbonate or bi-carbonate has been shown by the result of the determination of permanent hardness (q. v.).

Determination of the Permanent Hardness of Water.

Principle of the Method.—If sodium carbonate is added to water containing calcium and magnesium salts, the sodium carbonate reacts with the alkaline earths precipitating the calcium and magnesium carbonates. The Na_2CO_3 thus consumed is therefore a measure of these salts.

Apparatus.—Evaporating dish (11 cms.), 100 c.c. pipette, stirring rod, beaker, burette, steam bath, funnel, filter paper.

Reagents.—N/10 HCl solution, N/10 Na_2CO_3 solution, methyl-orange (.02 grs. per 100 c.c.).

Details of Procedure.—Measure by means of a pipette 100 c.c. of the water to be examined into a clean evaporating dish. Add a drop or two of methyl-orange and then from a burette 25 c.c. N/ 10 Na_2CO_3, or an amount sufficient to make the water strongly alkaline. Evaporate to dryness on the steam bath, then take up with a small amount of distilled water. The solution should show a distinct yellow color. If this is not the case, sufficient Na_2CO_3 was not added in the first place, consequently more must be added and the evaporation repeated. If the yellow color is obtained, filter off the insoluble calcium carbonate, wash the precipitate and paper with distilled water until free from alkalinity. Titrate the excess of alkali in the filtrate with N/10 HCl until the characteristic color for methyl-orange in acid solution is obtained. The difference between the amount of alkali added and the amount found in the titration is the amount of Na_2CO_3 used in precipitating the calcium and magnesium salts, which produce the permanent hardness.

Calculation of Result.—The calculation is made in the usual manner. Hardness is expressed in parts per million $CaCO_3$.

Volumetric Determination of Free Carbonic Acid.

Principle of the Method.—The principle of the method depends upon the following reactions:

a. $CO_2 + Na_2CO_3 + H_2O = NaHCO_3$.

b. $Na_2CO_3 + HCl = NaHCO_3 + NaCl$.

Since phenolphthalein is neutral toward bicarbonate, it may here be chosen for indicator.

Apparatus.—250 c.c. beaker, stirring rod, 100 c.c. pipette, 2 burettes.

Reagents.—N/10 Na_2CO_3 and N/10 HCl solutions, phenolphthalein, *neutral* alcohol.

Details of Procedure.—By means of the pipette measure into the beaker 100 c.c. of the water to be tested. Add two or three drops of phenolphthalein, then 10 to 15 c.c. of the *neutral* alcohol. From one of the burettes run into the water a known volume of the N/10 Na_2CO_3 solution sufficient to give a decided red color to the indicator. Now determine the excess of alkali added by titration with N/10 HCl. During this titration it is necessary to keep the solution rotating rapidly, otherwise local excess of acid might carry the reaction to the point of releasing carbon dioxide from the bicarbonates.

Calculation of Results.—By reference to reaction (b) under "Principle of the Method," it is noticed that when titrating normal carbonates with acid, phenolphthalein being the indicator, only half the quantity of acid required for complete neutralization is consumed. Consequently, in determining the excess of Na_2CO_3, the volume of HCl used in the titration must be doubled in order that it read in terms of normal carbonate. When the number of c.c. Na_2CO_3 in excess has been found, subtract it from the total number added. The remainder, of course, must be Na_2CO_3 equivalent to the acidity of the water.

The Effect of Iron in Water Used for Laundry Purposes.

Outline of Procedure.—In order to observe the effect of iron in water used for laundry purposes, fabrics are washed in water containing ferrous bicarbonate and the results noted.

Apparatus.—Balance and weights, watch glass, large casserole, piece of clean white cloth, 2 test-tubes with corks to fit.

Reagents.—Water containing free carbonic acid and dissolved air, solution containing ferrous bicarbonate approximately 50 parts per million, soap or soap solution.

Details of Procedure.—(a) Weigh out 0.13 to 0.16 grams of ferrous sulphate, and 0.14 to 0.16 grams of sodium bicarbonate ($NaHCO_3$). Dissolve first one then the other in a liter of aerated distilled water, through which CO_2 has been bubbled for two or three minutes. The following reaction then takes place:

$$FeSO_4 + NaHCO_3 = Fe(HCO_3)_2 + Na_2SO_4.$$

Transfer the bulk of this solution to the casserole. Soap the white cloth in exactly the same way as is done in washing process, then allow it to stand in the water over night. After standing, remove the cloth and examine carefully. (?).

(b) Take two test tubes, fill one, half full of distilled water and the other, half full of the water containing iron. Into each introduce a few scraps of soap, then stopper and shake. After

a few minutes compare the colors of the liquids in the two tubes. (?).

QUESTION.—Show by reactions how it is possible to obtain iron rust stains on fabrics washed in water containing ferrous bicarbonate.

Method for the Determination of Iron in Water.

PRINCIPLE OF THE METHOD.—Water is freed from organic matter. The iron is oxidized to the ferric state, then converted to thiocyanate, this being recognized by its characteristic red color. In order to make the determination quantitative, the color produced by the ferric thiocyanate from the water being examined, is compared with colors obtained by similar procedure from waters containing known amounts of iron.

APPARATUS.—Evaporating dish (11 cms.), 100 c.c. pipette, steam bath, 2-100 c.c. Nessler tubes of equal diameter, 100 c.c. graduated cylinder and siphon, liter flask.

REAGENTS.—HCl (1-1), $KMnO_4$ solution (5 grams per liter), KCNS solution (20 grs. per liter), standard ferric solution prepared by dissolving 0.1 gr. of pure iron wire in a little HCl, adding a few drops of $KMnO_4$, then diluting to a liter.

DETAILS OF PROCEDURE.—Measure into the evaporating dish 100 c.c. of the sample of water by means of the 100 c.c. pipette, and add 5 c.c. of HCl, then evaporate to dryness on the water bath. When the residue is absolutely dry, ignite the contents of the dish over a small flame of the Bunsen burner until the organic matter is destroyed, indicated by the disappearance of all carbonaceous matter. Now add 5 c.c. of HCl (1-1), dilute slightly, always keeping in mind that the total volume of the solution must never exceed the capacity of the Nessler tube; filter and wash. Add $KMnO_4$, a few drops at a time, until the slight pink color thus obtained disappears in not less than 5 minutes, and then add 10 c.c. of the KCNS solution. Rinse into a 100 c.c. Nessler tube, dilute to the mark, and mix thoroughly. Next prepare in an exactly similar manner another tube, using distilled water and 10 c.c. of the standard ferric chloride solution. Compare the colors of these two by looking through the depth of the liquids. By means of the glass siphon of narrow bore tubing, siphon out a portion of the contents of the tube having the deeper tint. Compare the tints again and continue the siphoning cautiously until the tints are of equal intensity.

Calculation of Results.—When the tints are of equal intensity, there are equal quantities of iron in each tube. Measure the number of c.c. of the standard solution and calculate the weight of iron contained therein, knowing the quantity of iron contained in the original 100 c.c. of the standard. This is the weight of iron in the quantity of solution in the second tube. Measure the number of c.c. contained in the second tube and calculate, if necessary, the quantity of iron in 100 c.c. of the solution. This is the weight of iron contained in 100 c.c. of the water under examination. Express it in parts per million.

Errors.—It is necessary that the preparation of the color from the standard and the unknown water be made at exactly the same time and under the same conditions, for the color is apt to change with time. The Nessler tubes must be of equal diameter. Why? Rust from iron utensils, etc., must not find access to the vessels used.

Coagulation of Clay and Mud.

Principle of the Method.—If a flocculent precipitate is formed in water containing clay or mud in suspension, this precipitate will settle on standing, and in doing so will carry the suspended material with it, thereby clarifying the water.

Apparatus.—A tall beaker or cylinder.

Reagents.—Aluminum sulphate, calcium hydroxide solution.

Details of Procedure.—Fill the beaker or cylinder two-thirds full with turbid water (water having clay or mud in suspension), add approximately 0.5 grams of powdered aluminum sulphate $Al_2(SO_4)_3$. Allow to stand until the salt has a chance to dissolve. Now add, without stirring, 10-15 c.c. of lime water $Ca(OH)_2$. Allow the water to stand and note the results.

Questions.—Write the reactions on which the principle of this experiment is based.

What material removes the suspended matter?

What becomes of the secondary product of the reaction?

Some Practical Questions.—1. From the knowledge obtained from the foregoing experiments, show by reactions the

effect of each of the following upon hard waters: Borax, washing soda, caustic soda, alum.

2. Calculate the quantity of any two of the above salts necessary to soften the water you analyzed.

3. Why is iron an objectionable ingredient in water used for laundry purposes?

4. What would you infer if in the determination of permanent hardness you found in titrating back the excess of alkali exceeded the total amount added? How would this figure affect the figure obtained for temporary hardness?

5. State briefly the effect of iron in water used for washing. Calculate the parts per million of iron in the water used in the experiment, "THE EFFECT OF IRON IN WATER USED FOR LAUNDRY PURPOSES."

B. SOAP.

Preparation of Soap (Hard).

PRINCIPLE OF THE METHOD.—Soap is prepared according to the following chemical equation:

$$(C_{17}\ H_{35}\ COO)_3\ C_3H_5 + 3Na(OH) = \\ 3C_{17}\ H_{35}\ COONa + C_3H_5(OH)_3.$$

REAGENTS.—Sodium hydroxide, fat or oil, salt.

DETAILS OF PROCEDURE.—Dissolve 15 grams of caustic soda in 120 c.c. of water. Place 60 grams of tallow in the casserole and pour half the lye solution upon it. Add 50 to 60 c.c. of water and boil for three-quarters of an hour. From time to time replace the water lost by evaporation. After boiling the stated time, add the remaining 60 c.c. of the lye solution and boil for an hour more. Water should again be added as in the early stages of the boiling, but finally the volume may be allowed to diminish one-third. At this stage, add 20 grams of sodium chloride, boil again for ten minutes and then allow to cool. The soap will rise to the top of the solution. Remove the cake, press out the contained liquid, dry between filter papers and then examine.

NOTE.—Keep this sample of soap for future use. At some time determine its quality.

Hydrolysis of Soap.

Principle of the Experiment.—Water hydrolyzes soap as follows:

$C_{17}H_{35}$ (COONa) + H_2O = $C_{17}H_{35}$ COOH + NaOH.

Now, since sodium stearate is a highly ionized substance, while the free acid is hardly ionized at all, the result of the interaction of the water with the sodium salt must be the production of hydroxidion (OH) in the solution.

Details of Procedure.—To two or three grams of soap add 50 c.c. of alcohol and heat in a beaker on the steam bath until all the soap is in solution. At this point cool, and then add five to ten drops of phenolphthalein. Dilute slowly and observe the results. (?).

A Study of Soap Solutions.

Object of the Experiment.—To study the tendency of aqueous soap solutions, to wet oil or oily substances and to emulsify them.

Reagents.—Neutral or red litmus paper, kerosene, dilute lye, soap solution (water), pure water.

Details of Procedure.—Cut a piece of litmus paper 6 cms. larger than the diameter of a beaker. Place the paper over the mouth of the beaker, draw it down on the sides until it is tight like the head of a drum and then secure it with a rubber band. Add enough kerosene to oil the entire surface of the paper. Near one edge of the head allow a single drop of lye to fall from a stirring rod. In another place repeat with a drop of soap solution, while in a third add a single drop of cold water. Note the results. (?). Allow to stand for some time and again examine.

Questions.—1. In washing, is it the lye formed by hydrolysis of the soap or the soap itself, that combines with the oil and grease that is present?

A Study of Soap Solutions (Continued).

Object of the Experiment.—First, to show the ease with which soap solutions penetrate oils. Second, to show that pure water has not the same property.

Apparatus.—500 c.c. beaker, glass tubing 4 mm. in cross section, large rubber stopper (one hole).

Reagents.—Soap solution.

Details of Procedure.—Seal one end of a piece of glass tubing, then cut it about four centimeters from the sealed end. Place the sealed end in the hole of the rubber stopper, the stopper thereby acting as a small stand. Fill the glass tube with cottonseed oil, then place the whole in the beaker. Add water carefully by pouring the water carefully down the side of the beaker until the top of the tube is covered by at least an inch of water. Observe the effect if any of the water upon the oil in the tube. Now add strong soap solution, 5 c.c. at a time, and observe after each addition. (?).

Question.—What aid is given by this experiment in answering the question, What makes soap and water cleanse oily fabrics?

A Study of Soap Solutions (Continued).

Object of the Experiment.—To compare the surface tensions of soap solutions and water by means of "capillary rise."

Apparatus.—Two capillary tubes of the same bore, two small crystallizing dishes, a centimeter rule, iron stand with two clamps.

Reagents.—Soap solution, pure water.

Details of Procedure.—Place some soap solution in one of the crystallizing dishes. Fill the second with pure water to exactly the same height as the first. Place a capillary tube in each of the iron clamps attached to the iron stand, then lower one into the pure water and the other into the soap solution. Note the results. Allow the tubes to stand until there is no increase in the height of either column. Now measure the capillary rise in each case and compare.

Question.—What is the relation of the height of the column to the surface tension?

A Study of Soap Solutions (Continued).

Object of the Experiment.—The object of the experiment is to measure the relative sizes of drops formed, in soap solutions of various strengths, by an oil, as e. g., kerosene. It is possible by this method to grade soaps according to their detergent powers.

Apparatus.—One tall beaker (12 cms.), glass tubing 4 mm. in cross section, iron stand and clamp, Bunsen burner.

Reagents.—Kerosene, soap solutions.

Details of Procedure.—Heat one end of the glass tubing in the blue flame of the Bunsen burner until the bore is only the thickness of a hair, then flatten it out as shown in the sketch, at C. Now bend the tubing until it is in the shape shown. Cut the limb A-B, 25 to 26 cms. in length. Four centimeters from the end "A" make a scratch "D" with the triangular file, and another "E" 8 cms. below the first.

Prepare 500 c.c. of an N/10 sodium oleate solution and prepare from this by dilution 500 c.c. N/20 sodium oleate, from the N/20 prepare by dilution N/40, and from the N/40 prepare N/80.

Place a portion of each of these soap solutions in suitable beakers to the same height in each case. Also provide a beaker containing pure water. Fill the dropper with standard kerosene and clamp it in a vertical position in one of the beakers, the bend resting on the bottom of the beaker. Allow the kerosene to flow and count the drops as they rise from the orifice "C," while the liquid in the main limb passes from "D" to "E." Repeat three times and average the results.

Repeat the experiment with each of the soap solutions mentioned above and with pure water.

Plot the results in the form of a curve, using the number of drops as ordinates and the concentrations of the solutions as abscissae.

Repeat the above, using several commercial soaps.

Calculation of the Result.—The ratio (drops given by soap under examination. Drops given by standard soap of the

same strength) is a measure of the detergent power.

ERRORS.—Beakers must be filled to the same height in every case, and the temperature must be the same.

NOTE.—Count the drops formed in skimmed milk, soap bark solution and any other foaming liquids.

Determination of Moisture (Direct Method).

APPARATUS.—Aluminum dish, weights.

DETAILS OF PROCEDURE.—The sample is obtained by cutting the sample cake of soap in half, then shaving thin layers from the freshly exposed surfaces. Care should be taken to cut entirely across the surface, for the inside of the cake might have a different moisture content than the outer layer.

Accurately weigh the aluminum dish. Place about two grams of the finely shaved sample into the dish and weigh again quickly to nearest centigram. The difference between the two weights equals the weight of sample taken for the determination.. Heat in the air oven at a temperature 105 degrees for two hours, allow to cool in a desiccator and weigh again. Repeat the heating for a half hour, cool and weigh as before. These weights should be constant.

CALCULATION OF RESULT.—Per cent. moisture = 100 (Loss of weight due to evaporation)/(Original weight of sample).

NOTE.—This method is not absolutely accurate, due to the possibility of the loss of some volatile oils during the heating.

Determination of Moisture.

PRINCIPLE OF THE METHOD.—By heating soap in an anhydrous solvent of high boiling point, the moisture may be expelled while the solid soap is held in solution.

APPARATUS.—Porcelain evaporating dish (8 cms.), balance and weights, Bunsen burner, asbestos gauze, thermometer.

REAGENTS.—Oleic acid or paraffin.

DETAILS OF PROCEDURE.—Place about 8 to 12 grams of oleic acid in the evaporating dish and heat to 120 degrees until all the water is expelled. The temperature is determined by suspending a thermometer in the acid. Regulate the height of the Bunsen flame until it is just high enough to maintain the acid at the required temperature, 120 degrees. Keep the flame at this height until the experiment has been completed.

Allow the anhydrous acid and dish to cool in a desiccator and then weigh accurately. Now add a sample of soap obtained as directed above, equal to about one-third the weight of the acid, and weigh once more. Place the dish on the tripod over the regulated flame and heat until the soap is dissolved and the moisture expelled. (Do not have the thermometer in the liquid during this heating.) Cool in a desiccator and weigh.

Calculation of the Result.—Per cent. moisture = 100 (Loss of weight by evaporation)/(Weight of sample).

Note.—Paraffin may be substituted for oleic acid.

The heating, as a general rule, does not take more than fifteen minutes.

Determination of Alkali.

Apparatus.—Erlenmeyer flask (250 c.c.), steam bath, burette.

Reagents.—*Neutral* alcohol, phenolphthalein, N/10 acid.

Details of Procedure.—Treat a freshly exposed portion of the soap with a drop of phenolphthalein, and if no red color is produced, free alkali is not to be expected.

Accurately weigh a sample of the soap, say two grams, transfer it to an Erlenmeyer flask and dissolve in 100 c.c. of neutral alcohol. Filter if there is undissolved carbonate or other residue, wash with alcohol, add phenolphthalein and titrate with standard acid (N/10).

Calculation of the Result.—Express the free alkali as per cent. of sodium hydroxide.

Note.—If the alcoholic solution reacts acid with the indicator, reverse the procedure, titrate with standard alkali and calculate the resultant free fatty acid as oleic acid.

If the soap contains both free alkali and acid, this method is open to objection, for during the heating of the alcoholic solution, some of the free alkali may neutralize a portion of the free acid and thereby give unreliable results.

C. BLUING.

Prussian Blue.

Object of the Experiment.—First, to prepare Prussian Blue by double decomposition between potassium ferrocyanide and ferric salts, as:

$4\ FeCl_3 + 3\ K_4FeCN_6 = Fe_4\ (FeCN_6)_3 + 12\ KCl.$

Second, to examine the properties of the substance.

DETAILS OF PROCEDURE.—Weigh out 0.7 grams of potassium ferrocyanide and 0.5 grams of ferric chloride. Dissolve separately in 50 c.c. of distilled water. When each has entirely dissolved, add one to the other. (?).

Divide into two portions and filter one of them.

a. Transfer a portion of the precipitate to a test tube, add a few crystals of oxalic acid and warm. (?). Dilute and examine. (?).

b. Place a second portion of the precipitate in an evaporating dish and add lye. (?).

c. To a third portion of the precipitate in an evaporating dish, add an acid. (?).

Wash the second main portion several times by decantation with hot water and pour the washings through a filter. Test a portion of the filtrate for chloride. (?). Note the color of the filtrate. (?). When the decanted liquid begins to show a decided blue color, pour the precipitate upon the filter and continue the washing with hot water. Save the filtrate. Test the filtrate for chloride. (?).

QUESTIONS.—1. Of what practical value is the knowledge obtained in this experiment?

2. What name is given to solutions similar to that obtained by washing the second precipitate?

Properties of Ultramarine.

OBJECT OF THE EXPERIMENT.—To examine the properties of ultramarine.

DETAILS OF PROCEDURE.—Crush a "bluing ball" into small pieces. Place some of them in a test tube, add sufficient water to cover them, then enough acid to make the solution acid. Hold a filter paper wet with lead acetate or nitrate over the mouth of the tube. Heat if necessary. (?). Repeat, using sulphuric acid. (?). To a second portion of the crushed material, add sodium or potassium hydroxide. Test with the lead paper. (?).

QUESTIONS.—1. What is the stability of ultramarine toward acids and alkalies?

2. What is its solubility in water?

How would you rank it as a bluing? Give reasons.

3. What was the original source of ultramarine.
4. How is it prepared artificially?
5. In what form is it usually found when intended for laundry use?
6. What have you learned as to the stability of ultramarine?

D. BLEACHES.

Bleaching Action of Chlorine and Hypochlorites.

Object of the Experiment.—To prepare hypochlorous acid, sodium hypochlorite, javelle water and bleaching powder, and to examine each for its bleaching properties.

Apparatus.—Small beaker, chlorine generating flask.

Reagents.—Solution of sodium hydroxide (10 per cent.), hydrochloric acid, potassium permanganate or manganese dioxide, litmus paper.

Details of Procedure.—Pass chlorine gas into 100 c.c. of the 10 per cent. Na(OH) solution until it no longer gives a test for alkali with litmus. Examine this solution for bleaching properties by dipping into it colored fabrics. Add an excess of hydrochloric acid. Is there any evidence of decomposition? Note the odor. The solution is commonly known as javelle water. It contains sodium hydrochlorite (NaClO) and sodium chloride (NaCl). Write the reactions for its formation.

To about 50 c.c. of the javelle water, add 50 c.c. of 2N HCl and distil. Examine the distillate for bleaching properties. What does it contain?

Slake 25 grams of quick lime, avoiding excess of water. Saturate it with chlorine. What is it? To about 20 grams of the material suspended in water, add sodium carbonate until no further precipitation takes place. Filter the solution and compare it with the sodium hypochlorite prepared above. Write the equation.

Note.—The bleaching action of hypochlorous acid and hypochlorites depends upon the readiness with which they part with oxygen. Compare the decomposition of chlorine water in direct sunlight. (?).

A solution of chlorine in water may be quickly and conveniently prepared by the addition of hydrochloric acid to an aqueous solution of a per salt (e. g. $KMnO_4$).

Hydrogen Peroxide as a Bleaching Agent.

Object of the Experiment.—To examine the bleaching properties of hydrogen peroxide on feathers, hair and ivory.

Details of Procedure.—Fill a test tube two-thirds full of hydrogen peroxide. Add ammonium hydroxide, drop by drop, examining after each addition, until small bubbles appear on the side of the tube. Insert the material to be bleached into the solution and examine from time to time.

Questions.—1. Upon what does the bleaching action of the peroxide depend?

2. Did you notice any tendency of the bleached material to rot? If so, to what do you attribute the rotting?

REMOVAL OF STAINS FROM FABRICS.

Methods of Procedure.

Ink Stains.—Ordinary writing fluid consists of a solution of tannate of iron and an anilin dye, drawing inks contain finely divided carbon in suspension, indelible inks frequently contain silver nitrate, red inks are usually a solution of anilin dye.

Fruit Stains.—Due either to coloring matter naturally present or to colored products formed by oxidation.

Grease Spots.—Dirt, of course, accumulates on grease, causing a discoloration which cannot be removed till the grease is dissolved.

1. Physical Methods of Removal.

Chiefly of value with fresh stains.

Treat the article with a chemically inert, emulsifying detergent, as e. g., skimmed milk, soap bark, stale beer, gum solutions.

Take a piece of white linen or cotton having an ink stain on it and allow to soak for some time in skimmed milk. Try also some of the other substances mentioned above.

2a. Chemical Methods of Removal (Solvents).

This treatment requires a knowledge of the cause of the stain, as e. g., iron rust or iron ink.

Take a piece of white linen or cotton on which are rust stains. Immerse in a tepid solution of hydrochloric acid 2N, watching carefully till the stain is seen to be dissolved, then wash thoroughly till free from acid.

Take a piece of cloth which is stained with a grease spot. Treat the dry cloth with a fat solvent, such as carbon tetrachloride, chloroform, ether or gasoline. Carbon tetrachloride and chloroform have the advantage of not being inflammable.

2b. Chemical Methods of Removal (Bleaching).

This is useful in removing organic stains, as indelible pencil. Sulphur dioxide, hydrogen peroxide or chlorine compounds may be used.

Take a piece of white cloth on which are spots of red ink. Remove them with bleach. Try sulphur dioxide, ammonical peroxide of hydrogen and chlorine or javelle water.

PATENT DUST CLOTH.

Details of Procedure.—Place portions of a patent dust cloth in a Soxhlet extractor and extract with ether. Cautiously evaporate the ether and weigh the residual oil or grease. Calculate the grams of oil per square foot of dust cloth.

Endeavor to identify the oil by determining the specific gravity saponification equivalent, iodine number, viscosity, non-saponifiable matter. (Get special instructions for each of these determinations.)

CLEANSING AND POLISHING METALS.

Method of Procedure.—Metals are usually tarnished by a layer of oxide or sulphide. This is best dissolved by appropriate solvents, such as do not attack the metals to any appreciable extent. The polishing is accomplished by friction or pressure of a substance which is softer than the metal, and therefore does not abrade it, but polishes by causing an actual flow of the metal under the applied pressure, with the result that all the abrasions are filled in and a perfectly smooth surface results.

Ammonium hydroxide dissolves many metallic oxides, especially that of copper. Potassium cyanide is an excellent solvent for silver compounds, but is objectionable because of its poisonous character.

As polishing powders, there may be mentioned rouge (or finely divided iron oxide), whiting (finely divided chalk) and less common in any but technical work, alumina. The polishing is done with the wet powder.

II. AIR AND ITS EXAMINATION.

A. DUST.

Principle of the Method.—A quantity of dust is collected by exposing a glass plate to the air. It is then examined under the microscope.

Apparatus.—Glass microscope slides, microscope and accessories.

Reagents.—Solution of Canada balsam in alcohol.

Details of Procedure.—Carefully cleanse the slides. Paint them with the solution of Canada balsam and expose to the air for a definite time. Place the cover glass on the slide and examine under the microscope, endeavoring to identify the objects seen.

B. HUMIDITY.

Principle of the Method.—If a thermometer, whose bulb is kept wet with a film of water, be exposed to dry air or air containing less than enough moisture to saturate it, water evaporates from the thermometer, absorbing heat from the mercury and the glass, so that a lower temperature is recorded. The lowering of the temperature is a measure of the humidity of the air, the difference between the "dry" and "wet" bulb thermometer being a maximum in perfectly dry air and zero in saturated air.

Details of Procedure.—Read the several "wet" bulb thermometers with their associated "dry" bulbed ones, at regular (say half-hour) intervals, noting the conditions in the room with respect to ventilation, i. e., whether windows are up, number of people in the room, number of burners burning, etc.

Calculation of Results.—An empirical table gives, for each dry bulb temperature and difference between "wet and dry," the vapor pressure of the moisture in the air. Divide this by the vapor pressure for zero difference of "wet and dry," and we have the relative humidity. Calculate the relative humidity of the outdoor air when heated to the temperature of the room without the loss or addition of moisture.

Note.—The excess humidity of indoor air over that out of doors (when calculated to the indoor temperature), is a good measure of the pollution of the atmosphere by exhalations or combustion products.

C. CARBON DIOXIDE IN THE AIR (METHOD OF PETTENKOFER).

Principle of the Method.—A measured volume of air is exposed to a known volume of standard barium hydroxide solution, the carbon dioxide of the air being absorbed and precipitated as barium carbonate. The excess of barium hydroxide is determined by titration with standard acid.

Apparatus.—Large glass globe holding several liters, bellows, 2 burettes, pipettes (50 and 100 c.c.).

Reagents.—Dilute hydrochloric or oxalic acid such that 1 c.c. is equivalent to 0.5 c.c. carbon dioxide gas at zero and 760 mm. pressure, phenolphthalein solution, 400 c.c. beaker and stirring rod.

Details of Procedure.—By means of the bellows fill the large glass globe with air to be tested. Avoid contamination from the breath while taking the sample. Add 100 c.c. of the barium hydroxide solution by means of the pipette (do not blow out the pipette). Stopper the globe and allow to stand for some hours, shaking the contents about occasionally, so as to wet the walls.

In the meantime standardize the reagents. Prepare the acid by dilution of a stronger solution whose concentration is accurately known. Titrate measured portions of this acid with the barium hydroxide.

After several hours, draw out 50 c.c. of the solution from the globe and titrate the excess barium hydroxide with the acid.

Calculation of Results.—

50 c.c. barium hydroxide require	"a" c.c. acid
100 c.c. barium hydroxide require	2a c.c. of acid
50 c.c. barium hydroxide require (after exposure)	"b" c.c. acid
100 c.c. barium hydroxide require (after exposure)	2b c.c. acid

Therefore, c.c. CO_2 in the volume of air taken = (2a-b) 0.5 c.c. at zero and 760.

Per cent. CO_2 = 100(a-b)/(volume of air—100 c.c.).

III. HEAT.

A. TO MAKE WATER-GAS.

Principle of the Method.—When steam is lead over carbon or anthracite heated to a temperature approaching redness, the following reaction takes place:

$$C + H_2O = CO + H_2.$$

The mixture of equal volumes of hydrogen and carbon monoxide so obtained is known as water gas.

Apparatus.—Tube of hard glass or quartz about 6 ins. long, Bunsen burner, flask and tubes to serve as boiler, bell-jar of perhaps a liter capacity.

Details of Procedure.—Assemble the apparatus (see Instructor) and drive steam through the apparatus until the air is entirely flushed out. Place the glass bell-jar in position and heat the tube containing the coke or coal, until gas begins to collect in the bell-jar. Continue heating and passing the steam till the jar has become full of gas.

Note.—1. Observe the character of the flame of burning water gas.

2. Add 4 c.c. of gasoline to every liter of the water gas. Observe the character of the flame of the burning gas thus enriched.

B. BUNSEN FLAME.

Examine the construction of a Bunsen burner, noting the area of the gas orifice at the base of the burner. Arrange an apparatus to observe the volume of gas issuing per minute under the city pressure, and calculate the linear velocity with which the gas passes through the orifice.

What causes "striking back?" How may it be remedied?

When is a "Bunsen flame" hottest, when burning yellow, when blue but quiet, or when roaring?

Test the temperatures of the various parts of the flame with a platinum wire.

C. COMBUSTION A MUTUAL PROCESS.

Take an ordinary lamp chimney and fit it with a doubly bored cork at the base. Through the holes in the cork, pass two glass tubes, one straight of wide bore (3/16 to 1/4″) the second bent at right angles of somewhat smaller bore tubing.

Connect the bent tube with the gas supply pipe and light the gas as it issues from the glass tube. Turn quite low and introduce cork carrying tubes into lamp chimney. Turn up the gas flame so that it gradually displaces the air from the chimney, and note, that on bringing the ends of the tubes together while continuing to turn on more gas, that the flame passes from the "gas" tube to the "air" tube, and we have a very faint, non-luminous flame of air burning in gas.

D. TO COMPARE THE RATE OF COOLING OF A WHITE OBJECT AND A BLACK OBJECT UNDER IDENTICAL CONDITIONS.

Principle of Method.—Two test tubes as nearly alike as possible are fitted, one with a white muslin covering, the other with black, and filled each with equal quantities of boiling water or hot water, at the same temperature. A thermometer is placed in each and the temperature is read at minute intervals. We then plot a temperature-time curve for each and compare the two. (The experiment may, of course, be extended by the use of coverings of different materials, as linen, silk, wool, etc., and also fabrics of different colors.)

Apparatus.—Six-inch test tubes, coverings for the same, supports for the tubes, thermometer for each tube, perforated rubber stopper for each tube, watch with second hand, wash bottle containing boiling water, large beaker, Bunsen burner and tripod with gauze.

Details of Procedure.—Boil sufficient water in the beaker to submerge the tubes. Place the tubes in the beaker and note that the water filling each is at the same temperature. Withdraw the tubes simultaneously, dry quickly, insert in the coverings, place them in the support and commence to read temperatures, reading the tubes alternately (or in rotation if more than two), so that the temperature of each tube is read at minute intervals.

Continue until the temperature of the tubes has fallen to within five degrees of the room temperature.

CALCULATION OF RESULTS.—Plot the results to a convenient scale on cross-section paper, with temperatures as ordinates and time as the abscissae and note which cools the more rapidly.

NOTE.—It would be well to silver a tube and carry out the experiment in comparison with a plain tube.

E. PROXIMATE ANALYSIS OF COAL.

OBJECT OF THE EXPERIMENT.—By making tests to determine the quantity of moisture, volatile matter, coke, ash, sulphur and heat of combustion, an analyst may reach a definite conclusion as to the quality of the coal. The following methods are not for the determination of any definite chemical compounds, but their results give a basis for the comparison of coals.

DETAILS OF PROCEDURE.—(a) *Determination of Moisture.* Weigh exactly one gram of coal in a porcelain crucible, which has previously been heated, cooled in a desiccator and weighed. Place the crucible with its contents in an air oven heated at 104 to 107 degrees C for one hour. Cool in a desiccator and weigh. The loss of weight is equal to the moisture in the sample. Express the results as per cent. moisture. (Keep for use in the determination of ash.)

(b) *Volatile Combustible Matter.* Weigh a large crucible and lid, which has previously been heated and cooled in a desiccator. Weigh into it exactly one gram of coal, cover and heat with the full flame of the Bunsen burner for exactly seven minutes. Cool in a desiccator and weigh. In this determination, the following conditions are essential. The bottom of the crucible must be 6 to 8 cms. above the bottom of the Bunsen burner, which must have a clear blue flame fully 20 cms. high when burning free. The crucible must be protected from draughts during the heating. The upper surface of the lid must burn clear. The loss in weight less the moisture in the powdered coal is the volatile matter in the coal. Express the result as per cent. of volatile combustible matter.

(c) *Coke.* The residue in the crucible is coke.

(d) *Ash.* Place the crucible containing the coal from which the moisture has been expelled, upon its side on a pipe-stem

triangle. Heat at first with a very small flame, gradually increasing the heat as the combustion proceeds. Be sure to allow free access of air to the crucible. Continue until all black particles are burned. The per cent. of coke minus the per cent. of ash, gives the per cent. of *fixed carbon.*

(e) *Heat of Combustion by Calorimeter.* The details for this determination will be found in the printed pamphlet that accompanies the calorimeter.

Keep the contents of the bomb for the determination of sulphur.

DETERMINATION OF SULPHUR.—Transfer the contents of the bomb after the determination of the heat of combustion to a 400 c.c. beaker. Add 200 c.c. of distilled water. Wash the bomb carefully with distilled water and add the washings to the main solution. Acidify the solution with HCl and filter. Place the beaker upon a tripod, cover with a watch glass and heat to boiling.

Precipitate the sulphate by adding 10 c.c. barium chloride ($BaCl_2$) to the boiling solution. If a precipitate does not fall immediately, allow the solution to stand over night.

Filter on a weighed Gooch crucible. Dry, ignite and weigh the barium sulphate. Calculate per cent. sulphur in the coal.

F. DRY DISTILLATION OF BITUMINOUS COAL.

OBJECT OF THE EXPERIMENT.—Bituminous coal is heated without access of air and the products thus formed are examined.

APPARATUS.—Retort.

DETAILS OF PROCEDURE.—Fill a retort one-half full of soft coal. Connect the exit of the retort with a train consisting of an empty bottle, to collect any liquids that might distil over, a second bottle containing wash water and a gasometer constructed by the use of small beakers filled with water inverted in a pneumatic trough.

Heat the coal. Discard the first 200 to 250 c.c. of gas collected in the gasometer, for it will be air displaced from the apparatus. Continue the heating until no more gas is evolved.

Identify the products in the first bottle. (?).

Add a drop or two of litmus to the water in the second bottle. (?). Test for volatile alkali. (?). What is it?

Set fire to the gas in one of the beakers. What is the character of the flame?

Add lime water to the gas in another beaker. (?).

Drop a paper wet with lead acetate into a third sample of the gas, cover the beaker and examine the paper after a time.

To a fourth sample, add a weak solution of bromine water and shake.

To still another sample, add a small piece of phosphorous. (?).

Question.—What do you conclude as to the composition of illuminating gas?

G. PREPARATION OF PARAFFIN WAX.

Chill about 50 c.c. of paraffin oil in a salt and ice mixture. Note the separation of paraffin. Drain off the oil. Melt and freeze again. Repeat till the wax is quite free from oil, washing with a little benzine if necessary, to facilitate the removal of the oil.

Finally, heat with bone black, filter and chill. We should have a piece of white paraffin wax.

IV. FOOD MATERIALS.

A. BAKING POWDER.

1. Identification of the Acid Constituents.

Object of the Experiment.—To analyze qualitatively for the various acid constituents of baking powder and thereby determine the class to which the powder belongs.

Apparatus.—Beaker, stirring rod, test tubes, evaporating dish, filter.

Reagents.—Ammonium molybdate, cupric hydroxide, potassium permanganate, oxalic acid, barium chloride.

Details of Procedure.—(a) *The powder contains an insoluble carbohydrate (starch) as filler.* Place about 5 grams of the powder in a beaker and cover with cold water. After the first violence of the action has subsided, stir so as to facilitate solution of all the soluble constituents. Allow to subside, then pour through a filter.

The Residue will contain:

1. Starch.
2. Secondary calcium phosphate.
3. Aluminum hydroxide.

Examine it as follows: Treat with cold dilute nitric acid and filter from the insoluble starch. Place a small portion of the nitric acid solution in a test tube, add an excess of ammonium molybdate; warm, shake, and look for the appearance of a canary yellow pulverulent precipitate of ammonium phospomolybdate, which indicates the presence of phosphates.

If phosphates are absent, to the remainder of the nitric acid solution add ammonium hydroxide; the formation of a white precipitate of aluminum hydroxide indicates alum in the powder. (If this test is found to be positive, apply the test for the detection of aluminum compounds, found at the end of the experiment.)

If phosphates are present, treat the remainder of the nitric acid solution with an excess (2 grams) of pure tin and 5 c.c. nitric acid. Evaporate to dryness, take up with nitric acid and water, allow to subside in a cylinder, filter from the insoluble meta-stannic acid and stannic phosphate, and test the filtrate for the presence of alumina by means of ammonium hydroxide as above.

Boil a portion of the RESIDUE with water, cool and add a solution of iodine. A blue color indicates starch.

The FILTRATE will contain:

1. Sodium potassium tartrate.
2. Secondary sodium phosphate.
3. Sodium sulphate.

Examine it as follows: Precipitate in a test tube a small quantity of cupric hydroxide by the addition of a solution of sodium hydroxide to one of cupric sulphate. Add to this a portion of the solution to be tested for tartrates and warm gently. Observe whether the cupric hydroxide dissolves with the production of a deep blue solution, which does not precipitate on boiling.

Place another portion of the solution in an evaporating dish and evaporate to dryness. Heat the residue. If it blackens with intumescence, emitting odor of burned sugar, we may assume the presence of tartrates.

Take another small portion of the FILTRATE and add a strong solution of potassium permanganate and boil, adding more permanganate if the solution bleaches out. Finally dissolve any brown precipitate by the addition of a few crystals of oxalic acid. Now test for the presence of phosphates by means of ammonium molybdate as above.

Take another portion of the FILTRATE, acidify with hydrochloric acid and add barium chloride. A white precipitate of barium sulphate indicates the presence of sulphates.

(b) *The powder contains a soluble carbohydrate (lactose) as filler.* Place about 5 grams of the powder in a beaker and

cover with water, stir to facilitate the solution and filter from the insoluble matters.

The RESIDUE will contain:

1. Secondary calcium phosphate.
2. Aluminum hydroxide.

Examine it as above.

The FILTRATE will contain:

1. Lactose.
2. Sodium potassium tartrate.
3. Sodium sulphate.
4. Secondary sodium phosphate.

Examine it as follows: Evaporate to dryness and heat till thoroughly charred. Extract with water. Test the filtrate for carbonates, by looking for effervescence upon the addition of acid. Their presence indicates tartrates in the powder. Test also for phosphates and sulphates as described above.

Boil a small portion with Fehling's solution. A red precipitate indicates lactose.

Detection of Aluminum Compounds in Baking Powders (Applicable in the Presence of Phosphates).

Ash about two grams of the material in a thin porcelain dish. Extract the residue with boiling water and filter. The filtrate will contain the alumina if present in the form of sodium aluminate. Add to the filtrate sufficient ammonium chloride to give it a distinct odor of ammonia. Alumina is indicated by the formation of a white precipitate of aluminum hydroxide.

$$Na_2Al_2O_4 + NH_4Cl + H_2O = 2Al(OH)_3 + 2NH_4(OH) + 2NaCl.$$

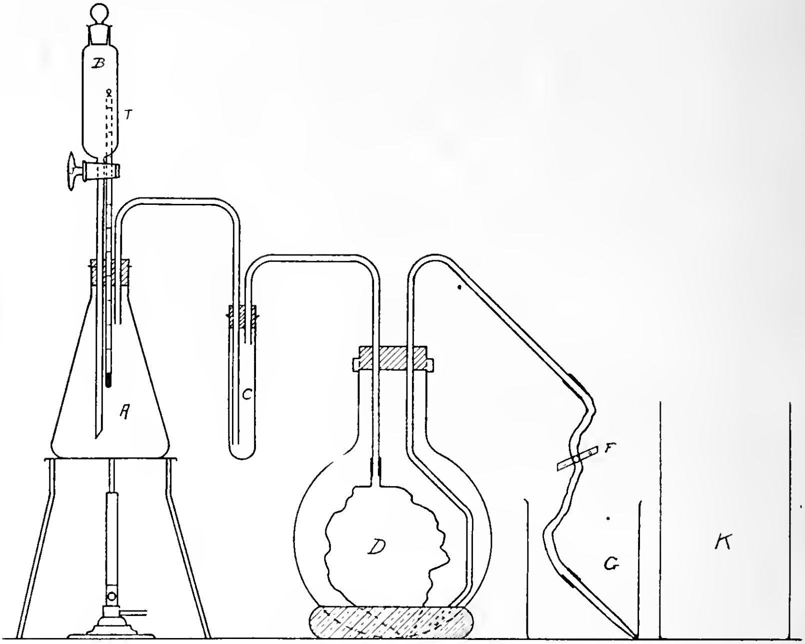
2. Detection of Ammonium Salts.

Place about two grams of baking powder in a large test tube, add an excess of a solution of sodium hydroxide and warm. Notice the odor and hold a piece of wet neutral litmus paper in the fumes and observe whether it turns blue.

3. Determination of Total Carbon Dioxide.

PRINCIPLE OF THE METHOD.—The baking powder is treated with acid, which liberates the carbon dioxide gas. The volume of the gas is measured by the displacement of water. Its weight is calculated from this volume, knowing the temperature and pressure.

Apparatus.—Special apparatus according to the following sketch:



Weights, 50 c.c. pipette, flat pan scales.

Details of Procedure.—Assemble the apparatus as shown in the sketch. Weigh the beaker "C" to the nearest decigram. Weigh from 10 to 15 grams of the baking powder to the nearest centigram. Clean and dry the Erlenmeyer flask "A," and introduce the weighed powder into it, without loss. Place the flask in position and open the stopcock "J" and then pinch clamp

"F" to equalize the pressure. It is necessary that the outlet tube at the end of the apparatus be completely filled with water. Close "F" and then "J." Drop exactly 50 c.c. of dilute sulphuric acid into the dropping funnel "B." It is absolutely necessary, of course, that all joints be air-tight. Place the weighed beaker "G" in position. Read the thermometer "T" and the barometer. Drop the acid in the tap-funnel into the flask by careful control of the stopcock "J." It is necessary that every drop of acid enter the flask, but no air, therefore, close the stopcock the instant the last portion of the acid enters the bore of the stopcock. When all the apparent action in the flask has ceased, boil gently for five minutes. Allow the apparatus to return to room temperature. This may be hastened by surrounding the flask "A" with cold water contained in the glass jar "K," until the thermometer "T" records the original temperature, then replacing "K" by a similar jar containing water at room temperature.

Now bring the liquids in the globe "E" and beaker "G" to the same level. After a few moments close "F," while the levels are the same. Remove the beaker "G" and weigh to the nearest decigram.

Calculation of the Result.—Let "W" grams of water be the amount displaced at the temperature "T" and the pressure "P," then we have (W-50) c.c. of carbon dioxide at "T" degrees cent., and "P" millimeters mercury pressure saturated with moisture. "A" is the aqueous tension at a temperature "T."

Per cent. carbon dioxide $= (W-50) \times 273 \times (P-a) \times .00198 \times 100/(273 + t)$ (weight of baking powder).

Carbon Dioxide Available in the Cold.

Principle of the Method.—A weighed quantity of baking powder is acted upon by water for a definite time at room temperature, the CO_2 being allowed to escape into the air. The residual CO_2 is then determined as under total carbon dioxide.

Apparatus.—As in determination of total carbon dioxide.

Details of Procedure.—Weigh 20 to 30 grams of baking powder to the nearest decigram. Transfer it to the flask "A" without loss, and add 10 c.c. of water for each gram of powder. Allow to stand for twenty minutes. Connect up the apparatus and proceed exactly as before with the determination of CO_2.

CALCULATION OF RESULT.—Calculate exactly as in the case of total CO_2.

4. Carbon Dioxide Available at 100 Degrees Centigrade.

PRINCIPLE OF THE METHOD.—A weighed quantity of the powder is treated with a definite amount of cold water, then heated to boiling and the carbon dioxide allowed to escape in the air. The residual carbon dioxide is then determined as described before.

APPARATUS.—The same as before.

DETAILS OF PROCEDURE.—Weigh 20 to 30 grams of the powder as before and treat with water in the proportion of 10 c.c. per gram of powder. After effervescence has ceased, heat to boiling for five minutes. Cool to room temperature, connect with the apparatus and proceed as before with the determination of the carbon dioxide.

CALCULATION OF THE RESULT.—The same as before.

5. Determination of Starch.

PRINCIPLE OF THE METHOD.—The baking powder is treated with cold dilute acid, whereupon all the components of pure baking powder, with the exception of starch, are dissolved. The insoluble starch is then separated from the soluble salts and determined.

APPARATUS.—Measuring cylinder, prepared Gooch crucible and filtering apparatus.

REAGENTS.—Hydrochloric acid (3 per cent. solution), alcohol, ether.

DETAILS OF PROCEDURE.—Weigh about one gram of the powder to the nearest milligram. Transfer to a clean 200 c.c. beaker and treat with 150 c.c. of the cold three per cent. acid, stirring for fifteen minutes. Filter through a Gooch crucible. Wash with water until neutral, and then follow with 15 c.c. of alcohol and finally with 15 c.c. of ether.

Dry the crucible and its contents in the air oven at 110 degrees for two hours. Cool in a desiccator and weigh. Place the crucible over a small flame of the burner for five minutes. Gradually increase the heat until it is sufficient to burn off the

starch. When this step is accomplished, cool in the desiccator and weigh.

Calculation of the Result.—Per cent. starch = 100 × loss of weight on ignition/weight of sample.

6. Determination of Lactose Used as Filler.

Principle of the Method.—The lactose is dissolved from the powder by treating with water and then determined in the solution by the use of Fehling's solution.

Apparatus.— 250 c.c. graduated flask, 50 c.c. pipette.

Reagent.—Fehling's solution.

Details of Procedure.—Weigh 5 grams of the powder. Transfer the weighed sample to a small beaker and treat with 150 c.c. of distilled water for five minutes. Filter if necessary, allowing the filtrate to run into a 500 c.c. graduated flask. Wash with distilled water until the volume of the water reaches the graduation mark on the flask. Mix thoroughly, draw off 100 c.c. (pipette) and determine the lactose therein as directed under milk.

Calculation of the Result.—Same as under milk.

B. MILK.

1. Sampling.

Before taking a sample for analysis, mix the milk thoroughly by pouring it back and forth from one container to another.

2a. Determination of the Specific Gravity (Pyknometer).

The weight of a definite volume of milk at 15 degrees is divided by the weight of an equal volume of water at the same temperature.

Apparatus.—Pyknometer, balance and weights, thermometer, two dry beakers.

Details of Procedure.—Weigh the carefully cleaned and dried pyknometer to the nearest centigram. Fill with milk, the temperature of which has been adjusted to 15 degrees, being careful to expel all air bubbles. Weigh again to the nearest centigram. Wash the pyknometer thoroughly and fill it with distilled water at 15 degrees. Weigh again to the nearest centigram.

Calculation of Results.—Specific gravity = (weight of milk)/(weight of water).

Note.—Normal milk should range between 1027 and 1033 specific gravity at 15 degrees.

2b. Determination of Specific Gravity (Lactometer).

Principle of the Method.—A floating body sinks in a fluid until the weight of the fluid displaced equals the weight of the body. From this it follows that the comparative depths of flotation of a definite body in various fluids measure the specific gravities of the fluids.

Apparatus.—Lactometer, cylinder, thermometer.

Details of Procedure.—Place the lactometer in the cylinder and pour in milk until it floats. Read the specific gravity on the stem. Take the temperature of the milk and correct the specific gravity by means of the table furnished with the instrument.

Note.—Normal milk should range between 1027 and 1033 specific gravity at 15 degrees.

3. Determination of Total Solids.

Principle of the Method.—If milk is heated to 100 degrees, the vapor pressure is sufficient to evaporate the water from it rapidly, though the temperature is not sufficient to decompose the organic matter.

Apparatus.—Flat aluminum dish, balance and weights, 10 c.c. pipette, steam bath, weighing bottle.

Details of Procedure.—Accurately weigh the aluminum dish to the nearest milligram. Place about 10 c.c. of milk in a weighing bottle and weigh to the nearest centigram. Pour the milk without loss into the aluminum dish. Weigh the bottle again to the nearest centigram.

Heat the dish on the steam bath for two hours. Dry it and cool in a desiccator. Weigh the dish with its contents to the nearest milligram.

Calculation of Results.—Per cent. total solids = 100 (weight of solid residue)/(weight of milk).

4. Determination of Ash.

Principle of the Method.—The milk is treated with nitric acid to assist in the oxidation of the organic matter, then evapo-

rated to dryness and finally ignited to burn off the carbonaceous material. The residue is the mineral matter or ash.

Apparatus.—Thin porcelain dish, Bunsen burner, tripod, triangle, desiccator, balance and weights.

Details of Procedure.—Carefully weigh the porcelain dish to the nearest milligram. Transfer to it from a weighing bottle a quantity of milk weighed accurately to centigrams (approximately 20 grams). Add 6 c.c. of nitric acid and evaporate on the steam bath. When dry, place on the tripod and heat *cautiously* to dull redness until the residue is perfectly white. Cool in a desiccator and weigh to the nearest milligram.

Calculation of Result.—Per cent. ash = 100 (weight of ash)/(weight of milk).

5a. Determination of Fat (Babcock Method).

Principle of the Method.—When concentrated sulphuric acid is added to milk the emulsion is destroyed, the milk is curdled, the casein subsequently dissolved. The fat globules coalesce in a melted state at the top of the liquid. By whirling in the centrifuge, the separation of the fat is hastened and rendered complete.

Apparatus.—Babcock centrifuge, Babcock test bottles, 17.6 c.c. pipette, 17.5 c.c. graduate, wash bottle containing boiling water.

Reagents.—Commercial sulphuric acid specific gravity 1.82 to 1.84, a liter of boiling water.

Details of Procedure.—Measure by means of the pipette 17.6 c.c. of the milk into a test bottle. Add one measure (17.5 c.c.) of the sulphuric acid. Mix thoroughly until the casein is thoroughly dissolved. Place the bottle in a pocket of the centrifuge. Balance the machine by placing another bottle similarly prepared in the opposite pocket. Fill the pockets with boiling water and whirl for five minutes. Add distilled water to the bottles until they are filled to the top of the graduation and whirl again for one minute. All the fat should now be found as a clear yellow continuous layer in the graduated neck of the bottle. If this is the case, read the upper and lower limits of the fat layer.

Calculation of Results.—The difference of the two readings is the per cent. fat.

Note.—The average per cent. of fat in milk is 3.5 to 4.0 per cent.

5b. Determination of Fat (Gravimetric Method).

Principle of the Method.—Fat is the only component of the dried milk residue soluble in anhydrous ether.

Apparatus.—Soxhlet extraction apparatus, paper thimble, balance and weights, tripod, wire gauze, Bunsen burner, large iron stand clamp, steam bath, oven, strip of fat free filter paper, 2½ by 22 ins., liter beaker.

Reagents.—Anhydrous ether.

Details of Procedure.—Roll the strip of filter paper into the form of a cylinder of a smaller diameter than the weighing bottle. Wrap around it a piece of fine copper wire allowing four or five inches of the free end of the wire to serve as a handle. Place about 5 grams of milk in the weighing bottle. Weigh accurately to the nearest centigram. Dip the paper coil into the bottle and allow all or as much of the milk as possible to be absorbed. Remove the coil and hook the copper wire over a glass rod, which is placed across the top of an empty beaker. Weigh the bottle again to the nearest centigram, place the beaker containing the coil on the steam bath and heat until the paper is dry, finishing in the oven, the temperature of which never exceeds 100 degrees C.

Weigh the receiving flask of the Soxhlet apparatus. Transfer the dried paper coil to the thimble of the extractor. Assemble the parts of the apparatus and add sufficient anhydrous ether down the top of the condenser to cause the syphon to act, then 30 to 40 c.c. in excess. Heat cautiously, regulating the flame to the proper height, so that the ether boils quietly. Extract for at least two hours. Evaporate the ether extract and washings in the flask by placing it on the steam bath and finally drying in an air oven at 100 degrees cent., while aspirating air through the flask. Cool and weigh.

Calculation of Results.—Per cent. of fat = 100 (weight of fat)/(weight of milk).

Sources of Error.—Avoid placing more milk in the weighing bottle than the paper can absorb, otherwise an error due to selective absorption may occur. Do not allow the milk soaked

coil to come in contact with any object lest a portion of the milk be lost.

If the coil is not thoroughly dry before extraction or the ether used is not anhydrous, water soluble substances (sugar) may be dissolved in addition to the fat.

6. Determination of Milk Sugar.

Principle of the Method.—The proteins of the milk are precipitated by adding copper sulphate solution, the fat being entangled with the precipitate. The soluble milk sugar is then determined in the supernatant liquid from the weight of copper it will reduce from Fehling's solution.

Apparatus.—500 c.c. graduated flask, 10 c.c., 25 c.c. and 50 c.c. pipettes, carbon filter tube, rubber-stoppered filter flask, suction pump, 50 c.c. graduated cylinder, balance and weights, Bunsen burner, tripod, 400 c.c. beaker, burette, air oven, stirring rod, wash bottle, desiccator.

Reagents.—Fehling's solution (A. Dissolve 34.639 grams of copper sulphate $CuSO_4 5H_2O$ in water, then dilute to 500 c.c.; B. Dissolve 173 grams of Rochelle salt and 125 grams of K (OH) in water and dilute to 500 c.c., use equal volumes of A and B, N/2 Na(OH), distilled water.

Details of Procedure.—Measure into the 500 c.c. flask by means of a pipette, 25 c.c. of milk, the specific gravity of which is known. Dilute to about 400 c.c. with distilled water. Add 10 c.c. (pipette) of copper sulphate solution ("A" solution of Fehling's. See above). Now add from a burette, 8.8 c.c. of N/2 Na(OH) solution. The solution should still retain an acid reaction. Dilute to the mark, mix well and allow to settle.

Prepare a Gooch crucible. (See Instructor.) Dry the Gooch in an air oven heated to 100 degrees. Cool in a desiccator and weigh.

Filter a quantity of the liquid containing the milk sugar into a *dry* beaker. Reject the first 100 c.c.

Place 50 c.c. of the *mixed* Fehling's solution (equal volumes of A and B in the 400 c.c. beaker and heat to boiling on the tripod. To the boiling solution, add 100 c.c. of the clear filtered solution containing the milk sugar. Boil for precisely six minutes. Filter immediately through the Gooch, wash two or three times by decantation with hot water, then transfer the entire precipitate to the Gooch. Remove the water by washing with alcohol and the alcohol by washing with ether. Dry in an air oven (after the odor of ether is gone) at a temperature not exceeding 100 degrees C. Cool in a desiccator and weigh.

Calculation of Results.—By means of the tables in Bulletin 107, Bureau of Chemistry, pages 48-49, find the equivalent of the Cu_2O in terms of lactose, then:

Per cent. lactose = (weight of lactose) × 5 × 100/25 (specific gravity).

7. Determination of Total Nitrogen.

Principle of the Method.—Milk is digested with concentrated sulphuric acid until all the nitrogenous substances are converted to ammonium sulphate and the ammonia subsequently distilled out with Na(OH) and caught in a measured excess of acid of known strength.

Apparatus.—Kjeldahl digestion flask and distillation apparatus, Bunsen burner, tripod, iron stand, clamp, burette, pipette (5 and 10 c.c.), Erlenmeyer.

Reagents.—C. P. H_2SO_4 specific gravity 1.84, saturated Na(OH), N/10 H_2SO_4, N/10 Na(OH) or K(OH), powdered K_2SO_4, phenolphthalein.

Details of Procedure.—Place in the Kjeldahl digestion flask 5 c.c. of milk (pipette). Add 10 grams of powdered potassium sulphate, then 20 c.c. conc. sulphuric acid. Digest over a small flame until the danger from foaming has passed, then raise the temperature till the acid is boiling and continue the heating until the contents of the flask are light straw-colored or colorless. Cool, add about 250 c.c. of distilled water. Add a little phenolphthalein and two or three pieces of pumice.

Assemble the apparatus, putting 50 c.c. (pipette) N/10 H_2SO_4 in the absorption flask. Add 50 c.c. saturated Na(OH) to the Kjeldahl flask. (The solution in the flask should be strongly alkaline.) Immediately replace the stopper and make sure that all joints are tight. Heat cautiously, finally raising to the boiling and continue until all the ammonia has passed over or until the bulb of the receiver is filled with liquid.

Remove the receiver, add methyl-orange and titrate the excess of H_2SO_4 with N/10 K(OH) or Na(OH).

Calculation of Result.—

1 c.c. N/10 H_2SO_4 = .014 grams nitrogen.
= .014 × 100/15.7 (since protein contains 15.7 per cent. N).
= .014 × 6.38.

Per cent. protein = V × .0839 × 100/5 (specific gravity), where "V" is the volume of N/10 acid neutralized by the ammonia.

8. Detection of Added Water (Zeiss Dipping Refractometer).

Principle of the Method.—When light enters a medium in which its velocity is either increased or diminished, the light is bent or refracted. A definite pair of substances produces a definite effect, that it has a constant refractive index.

Pure milk serum has a refractive index ranging from 40 to 42.5 on the arbitrary scale of this instrument. Added water lowers the refractive index.

Apparatus.—Zeiss immersion refractometer, thermometer, beaker, filter paper.

Reagents.—Twenty-five per cent. acetic acid (specific gravity 1.035).

Details of Procedure.—To 100 c.c. of milk, at a temperature about 20 deg. C, add 25 c.c. of the acetic acid and heat in a covered vessel on a water bath for twenty minutes at 70 degress C. Place the beaker in ice water for ten minutes and separate the curd from the serum by filtering through a dry paper. Transfer about 35 c.c. of the serum to one of the beakers of the Zeiss apparatus, adjust the temperature to exactly 20 degrees and take the refractometer reading.

Note.—A reading below 39 indicates added water, between 39 and 40, the sample is to be considered suspicious.

9. Detection of Formaldehyde.

Principle of the Method.—Milk containing formalin when heated with hydrochloric acid containing a trace of ferric chloride gives a characteristic violet color.

Apparatus.—Twelve cm. casserole, Bunsen burner.

Reagents.—Concentrated HCl, containing $FeCl_3$ in the proportion of 2 c.c. of a 10 per cent. solution of ferric chloride per liter.

Details of Procedure.—Place 10 c.c. of the milk to be tested in a casserole and add an equal quantity of the hydrochloric acid. Hold the casserole over a small flame of the Bunsen burner and heat slowly, almost to boiling. During the heating, give the casserole a rotary motion in order that the curd when formed, will be broken up. The presence of formaldehyde will be indicated by the formation of a violet coloration.

Note.—If the curd acquires a pink color immediately upon the addition of the acid, an azo dye is indicated (q. v.).

In making this test, first use a sample to which a trace of formalin has been added.

The use of formaldehyde can *never* be justified.

10. Detection of Boric Acid.

Principle of the Method.—Turmeric paper, wet with a solution of boric acid, gives, upon drying, a rose pink coloration.

Apparatus.—Nine cm. casserole, Bunsen burner, tripod, water bath, filter stand, two-inch funnel.

Reagents.—Turmeric paper, ammonium hydroxide, hydrochloric acid.

Details of Procedure.—Place 5 c.c. of milk in the casserole, make distinctly alkaline with Na(OH) and evaporate to dryness on the steam bath. Destroy the organic matter by ignition. Add 10 to 15 c.c. of water to the ash, then HCl, drop by drop, until the liquid is acid. Filter. Dip a strip of turmeric paper into the filtrate and dry on the outside of a test tube in which water is being boiled. A rose red color indicates boric acid. Ammonium hydroxide turns the paper dark green. Heating restores the original color.

Questions.—Why is it necessary to add the Na(OH) before the evaporation?

Why acidify the ash with HCl?

Does the change to dark green from the rose red turmeric paper, upon the addition of ammonium hydroxide, make this

If the curd is perfectly white, azo dye and caramel are absent. The color, if any, must be annatto.

a. Annatto is present.

Evaporate the ether solution and treat the residue with dilute Na(OH) solution. Pour the liquid on a wet filter paper. The fat is caught on the paper, while the annatto dyes it. Wash the fat from the paper with a stream of hot water. Allow the paper to dry. Annatto, if present, will impart an orange color to the paper. Add a few drops of $SnCl_2$ and it becomes pink in the presence of annatto.

b. Azo dye.

If the extracted curd is colored, add conc. HCl to a portion of it. If azo dyes are present, the curd becomes pink.

c. Caramel.

If the extracted curd has a brown color and neither azo dyes nor annatto are found, we may assume the presence of caramel.

Confirmatory test for caramel.

To 100 c.c. of milk, add an equal volume of alcohol and filter. Any caramel is in the whey. Add a solution of basic lead acetate and filter. Dry the precipitate (exclude all H_2S) and if the precipitate is then either white or pale straw color, caramel is absent; if more or less brown, caramel is present.

NOTE.—Azo dyes are more frequently used for this purpose than either of the other two.

12. Examination of Sediment in Milk.

Fill two sedimentation tubes with a sample of common milk. Whirl a few minutes in a centrifuge. If sufficient sediment is not obtained, decant the milk carefully, fill again with milk and whirl again. Repeat till sufficient sediment is obtained to permit of its macroscopic and microscopic examinations. Place on a slide and examine.

13. Development of Acidity.

Prepare six clean sterilized Erlenmeyer flasks. Add to each 50 c.c. of a sample of common milk with a sterilized pipette. Stopper each flask with cotton and titrate them successively with $N/10Ba(OH)_2$ and phenolphthalein at intervais of not less than 6

hours. Plot a curve showing the acidity as a function of the time.

14. Volatile Acids Developed on Souring.

Proceed the same as in the development of the acidity up to the point of titration. At this point acidify with sulphuric acid (see volatile acids in butter, p. 46), and distil off the volatile acids. Titrate with $N/10Ba(OH)_2$.

Plot the quantity of volatile acids produced as a function of the time.

Some Practical Questions.—1. What might you infer if two samples of milk should have the specific gravities: (a) 1036, (b) 1025? What other inference might be drawn from milk having a specific gravity of 1025?

2. What significance has low total solids?

3. What would you conclude if analysis showed:

(a) Total solids low, the ratio of solids not fat to fat about the same as in normal milk?

(b) Total solids and fat low, while solids not fat about normal?

(c) Total solids low, solids not fat materially reduced, and the ratio of solids not fat to fat also proportionately reduced?

C. EXAMINATION OF BUTTER.

1. Sampling.

If a large amount of butter is to be tested, the method by which the sample is obtained is of vast importance. To obtain a uniform sample, take a large number of small quantities from various parts of the butter until from 100 to 500 grams, according to the original bulk, are obtained. Add the portions together. Save a sample for microscopic examination. Melt the remainder of the sample in an Erlenmeyer at the lowest possible temperature. Allow the mass to cool. During the cooling, stopper the flask and shake it until it is sufficiently cool to prevent the separation of water and fat. Pour the butter into an Erlenmeyer so small that it just holds the sample. Stopper and then put in a cool place until needed.

2. Determination of Moisture.

Principle of the Method.—A sample of butter is heated to a temperature of boiling water until all the moisture is volatilized. The quantity is determined by the loss in weight.

Apparatus.—Aluminum dish (9 cms.), balance and weights, steam bath, desiccator.

Details of Procedure.—Accurately weigh the aluminum dish. Place about three grams of butter in this dish and weigh again. Heat the dish on the steam bath for one hour, dry it, cool in a desiccator, then weigh. Repeat the heating for another half hour, dry cool and weigh once more. Repeat the operation until two consecutive weighings show no change in weight.

Calculation of Results.—Express the loss of weight as per cent. of water.

Note.—Keep the material in the aluminum dish for the "Determination of Fat."

3. Determination of Fat.

Principle of the Method.—The method depends upon the fact that fat alone is dissolved from *dry* butter by anhydrous ether.

Apparatus.—The aluminum dish containing the residue from the Determination of Moisture, Gooch crucible, small wash bottle containing anhydrous ether, oven, desiccator, balance and weights.

Reagents.—Anhydrous ether.

Details of Procedure.—Treat the weighed contents of the aluminum dish with pure anhydrous ether. Pour all the washings through a dried and weighed Gooch, and finally transfer all the material to the Gooch. Wash the contents of the Gooch with ether till free from fat, then dry the crucible (after the odor of ether has disappeared), in an air oven at 100 degrees. Repeat the drying till the weight is constant.

Calculation of Results.—The loss in weight upon extraction with ether is the weight of fat.

Per cent. Fat = 100 (weight of fat)/(weight of butter).

Note.—Keep the Gooch and contents for the determination of ash.

Caution.—Be sure that no flames are in the vicinity when using ether. It boils at 37 degrees C and its vapor is inflammable.

4. Determination of Casein and Ash.

Principle of the Method.—The residue from the fat extraction which contains only casein and mineral matter is ignited. The casein is first carbonized, then the mass is incinerated, whereupon the casein is entirely consumed, leaving the mineral matter.

Apparatus.—Tripod, Bunsen burner, crucible and residue from fat extraction, desiccator, balance and weights.

Details of Procedure.—Cover the Gooch crucible containing the residue from the fat determination and heat carefully with a small flame, gradually increase the heat to a temperature just below redness. Remove the cover and continue the heating at the same temperature till the contents of the crucible are perfectly white. Cool in a desiccator and weigh.

Calculation of Results.—Per cent. casein = 100 (loss on ignition)/(weight of butter). Per cent. ash = 100 (weight of ash)/(weight of butter).

5. Determination of Salt.

Principle of the Method.—The salt is extracted from the butter by repeatedly washing the sample with hot distilled water. The quantity of salt in the aqueous solution is determined volumetrically with silver nitrate.

Apparatus.—Beakers, 1-100, 1-400 c.c., separatory funnel, wash bottle with boiling water, burette, stirring rod, pipettes, 1-10, 1-25 c.c., 1-250 c.c. graduated flask.

Reagents.—N/10 silver nitrate, potassium chromate solution.

Details of Procedure.—Weigh the 100 c.c. beaker accurately to the nearest centigram. Add from 5 to 10 grams of butter and weigh again. Add about 25 c.c. of boiling water and pour into the separatory funnel. Shake well and then allow to separate. Draw off the water into the 250 c.c. graduated flask and repeat the extraction with water till the graduated flask is almost filled to the mark. Cool the flask and the contents to room temperature, adjust to the mark with distilled water and mix thoroughly.

Take 10 c.c. of the salt solution in a beaker, add about 1 c.c. of K_2CrO_4 solution and titrate with N/10 silver nitrate solution to the production of a faint red tint in the place of the pure yellow color of the solution. From this preliminary trial, determine the most suitable volume of the salt solution that should be taken to yield an accurate result. Measure into the 400 c.c. beaker the determined volume of salt solution, add 1 c.c. of potassium chromate solution and titrate as before, remembering that the tendency is to overshoot the mark by adding too much silver nitrate solution; stop at the first departure from the pure yellow color. Make a duplicate titration if the quantity of the salt solution permits.

CALCULATION OF RESULTS.—Per cent. salt = 100 (c.c. N/10 silver nitrate per 250 c.c. salt solution × .00585)/(Grams of butter).

NOTE.—Salt usually present varies from 0.5 to 0.7 per cent.

6. Examination of the Melted Fat.

PRINCIPLE OF THE METHOD.—When fat is melted at a temperature of 50 degrees C, that separating from fresh genuine butter is clear, while that from process or renovated butter or oleo is more or less turbid. This is due to the fact that these latter fats contain a certain proportion of milk that has not been eliminated in the manufacture.

DETAILS OF PROCEDURE.—Observe the above in the preparation of the butter fat (q. v.).

7. Preparation of Butter Fat for Examination.

NOTE.—This may be done by one student preparing sufficient of the fat for the whole class.

Melt the butter (10 grams per student if working singly) in a sufficiently large test tube, heated in a bath of water at 50 degrees for two or three hours, until the water and curd have entirely separated from the fat. Pour the clear supernant fat through a dry filter paper. Keep the aqueous liquid at the bottom of the tube for the detection of boric acid, (q. v.). See "EXAMINATION OF THE MELTED FAT" (q. v.).

8. Determination of Volatile Acids.

PRINCIPLE OF THE METHOD.—The butter fat is saponified with sodium hydroxide, the soap thus formed decomposed with sulphuric acid, liberating the free fatty acids. The volatile acids are then separated from the fixed acids by distillation and measured by titrating the distillate with standard barium hydroxide.

Apparatus.—Erlenmeyer flask, 8 oz, distillation apparatus, receiver graduated at 110 c.c., Bunsen burner, tripod, wire gauze, funnel, burette, stirring rod, pipettes 25 c.c. and 100 c.c.

Reagents.—Standard barium hydroxide approximately N/10 hydrochloric acid, sulphuric acid 1-5, glycerol-soda solution (20 c.c. of a solution of NaOH, 1 gram solid to 1 c.c. of water, plus 180 c.c. glycerol, pumice, phenolphthalein (1 per cent. alcoholic solution).

Details of Procedure.—Weigh the Erlenmeyer flask to the nearest centigram. Add about 5 grams of fat prepared as directed above and weigh again to the nearest centigram. Measure into the flask 20 c.c. of the glycerol soda solution and heat the flask over the Bunsen flame till the fat is completely saponified; complete saponification being indicated by the contents becoming homogeneous, and bright and clear. Allow to cool and add 135 c.c. cold distilled water, avoiding the production of foam. When the soap has entirely dissolved, add 5 c.c. of the dilute sulphuric acid (1-5). Add a few pieces of pumice and distil regularly, so that 110 c.c. are collected in one half hour.

During the distillation, place the clear barium hydroxide solution in a burette. Place 25 c.c. N/10 HCl in a beaker, dilute slightly with water, add phenolphthalein and drop in the barium hydroxide till the end point is obtained. Repeat several times till the results are concordant. This standardizes the barium hydroxide.

Mix the distillate thoroughly, filter if necessary from any solid particles, take 100 c.c. of the clear liquid by means of a pipette and titrate with the standardized barium hydroxide, using 0.5 c.c. phenolphthalein as indicator.

Carry out a blank determination with the reagents, i. e., go through the operations above described, omitting the butter fat.

Calculation of the Result.—

Reichert-Meissl number = c.c. N/10 alkali required by volatile acids from 5 grams of fat.

= (c.c. N/10 Ba(OH)$_2$ × 1.1 ×5/(weight of fat).

Note.—The U. S. standard butter fat has a Reichert-Meissl number not less than 24. Oleomargarine or butterine has a Reichert-Meissl ranging from 0.5 to 5.5. This test is therefore used to detect the presence of foreign tissue fat in butter.

9. Detection of Boric Acid.

Principle of the Method.—The borax or boric acid is to be found in the aqueous liquid that separates from the butter on melting. The boric acid is then detected by the turmeric paper test as described under milk (q. v.).

Apparatus.—Test tubes, casserole, Bunsen burner.

Reagents.—Same as under milk.

Details of Procedure.—Take the aqueous liquid which separates in the preparation of the fat, q. v., and apply the test as described under milk.

10. Detection of Glucose.

Principle of the Method.—The soluble glucose is extracted from the butter by repeatedly washing with hot water, and its presence in the aqueous solution detected by means of Fehling's solution. (See the determination of lactose in milk.)

Apparatus.—Beakers, 1-1000 and 1-400 c.c., separatory funnel, wash bottle, tripod and burner.

Reagent.—Fehling's solution.

Details of Procedure.—Place 10 to 15 grams of butter in a small beaker. Pour a quantity of boiling water over the sample and when melted, pour into the separatory funnel. Shake, allow to separate, draw off the aqueous layer, heat to boiling and add Fehling's solution. A yellow or red precipitate indicates the presence of glucose.

Question.—How could the above procedure be made quantitative?

11. Foam Test.

Principle of the Method.—An unmelted fat, such as butter, foams when heated over a flame, whereas those that have been melted, such as lard, sputter noisily when so heated.

Apparatus.—Two small evaporating dishes, Bunsen burner, tripod.

Details of Procedure.—Heat three or four grams of butter in a small evaporating dish over a free Bunsen flame and note whether the fat foams quietly or sputters and crackles noisily. Repeat using a small quantity of oleomargarine or process butter.

Note.—This test may readily be carried on in the kitchen by

substituting a spoon for the evaporating dish and the flame of the gas stove for the burner.

12. Microscopic Examination of Butter, Process Butter and Oleo.

Principle of the Method.—Fats that have been melted contain crystals of fat, those which have not as butter are non-crystalline. Crystallinity can be detected by the effect of the material on polarized light, crystalline bodies giving a parti-colored field, while non-crystalline bodies give a uniform field.

Apparatus.—Microscope with polarizing attachment, slides, cover glasses.

Details of Procedure.—Place a small amount of the sample to be tested on a clean glass slide, press a cover glass over the fat and examine under the microscope with polarized light. Repeat, using samples of oleo, lard or beef drippings.

13. Detection of Artificial Coloring.

Note.—The artificial coloring of butter is quite common and is permitted by the laws of all the states.

At the present time, annatto and oil soluble azo dyes are practically the only colors used.

(a) *Detection of annatto.*

Principle of the Method.—The solubility of annatto in sodium hydroxide and the ability of filter paper to absorb the color from this solution are the principles upon which this detection depends. The final verification of annatto is by the reaction of stannous chloride upon it.

Apparatus.—One hundred c.c. beaker, funnel, filter paper.

Reagents.—Fat, dilute solution of sodium hydroxide, stannous chloride.

Details of Procedure.—Treat two or three grams of the melted and filtered fat (freed from salt and water) with warm dilute sodium hydroxide and after stirring, pour the mixture upon a warm wet filter, using to an advantage a hot funnel. If annatto is present, the filter will absorb the color so that when the fat is washed off by a gentle stream of water, the paper will be dyed straw color. It is well to pass the warm alkaline filtrate two or three times through the fat on the filter to insure removal of the color. If after dyeing the filter, the color turns pink on the application of stannous chloride solution, the presence of annatto is assured.

(b) *Detection of azo colors* (*Geisler's method*).

Principle of the Method.—Azo dyes will produce a pink or violet red coloration upon some varieties of fuller's earth.

Apparatus.—Porcelain plate.

Reagent.—Fuller's earth.

Details of Procedure.—Spread a few drops of the clarified fat upon the porcelain plate and add a pinch of fuller's earth. In the presence of various azo dyes, a pink to violet red coloration will be produced in a few minutes.

Note.—Some varieties of fuller's earth react much more readily than others, in fact, some do not react at all. It is, therefore, essential that the fuller's earth be previously tested.

D. OLIVE OIL.

1. Detection of Cottonseed Oil in Olive Oil.

Object of the Experiment.—To test an unknown sample of olive oil by the Bechi and Halphen methods in order to detect adulteration by the addition of cottonseed oil.

Apparatus.—Test tubes, beaker, tripod, Bunsen burner.

Reagents.—Bechi's reagent, prepared by dissolving 2 grams of silver nitrate in 20 c.c. of 95 per cent. alcohol free from aldehyde, adding 40 c.c. of ether and then made slightly acid with nitric acid.

Halphen's reagent is a mixture of equal volumes of amyl alcohol and carbon disulphide in which 1 per cent. of sulphur has been dissolved.

Details of Procedure.—(a) Bechi's test (Hehner's modification).

Mix a small amount of the oil in a test tube with half its volume of Bechi's reagent and immerse the tube in boiling water for fifteen minutes. The presence of cottonseed oil is indicated by darkening due to reduction of silver.

Halphen's test: Mix in a test tube a small quantity (5 to 10 c.c.) of the oil with an equal volume of Halphen's reagent.

Loosely stopper the tube with cotton and heat for fifteen minutes in a bath of saturated brine. A deep red or orange color shows the presence of cottonseed oil.

NOTE.—Heat a sample of cottonseed oil to a temperature of 250 degrees for ten minutes and then apply Halphen's test. (?).

2. The Effect of Light in Increasing the Acidity.

OBJECT OF THE EXPERIMENT.—Samples of olive oil are exposed to sunlight and the acidity is determined after exposures of different intervals.

APPARATUS.—Four glass-stoppered bottles 150 to 200 c.c. capacity, burette, 50 c.c. pipette.

REAGENTS.—Alcohol (*neutral*), phenolphthalein, N/10 Na(OH) or K(OH).

DETAILS OF PROCEDURE.—(a) By means of the pipette measure into each of the four bottles 50 c.c. of the oil to be examined. Give the pipette ample time to deliver the total 50 c.c. Record this. Allow the same period for drainage each time the pipette is used to deliver a sample of oil. Stopper each of the bottles and set them in the sunlight.

(b) Measure 50 c.c. of the oil (pipette) into a beaker, then add 50 c.c. of neutral alcohol. Add 10 to 15 drops of phenolphthalein and titrate with N/10 alkali. Record the result.

(c) After one week's time remove the stopper from one of the bottles, add 50 c.c. of neutral alcohol, 10 to 15 drops of phenolphthalein and titrate as before with N/10 alkali. Record the result.

(d) After an interval of two weeks repeat "c" with a second bottle. At three weeks' time determine the acidity of a third, and at the end of four weeks repeat the same with the last sample. Record the results.

CALCULATION OF THE RESULTS.—Record the acidity in terms of c.c. of N/10 acid per 100 c.c. of oil, also in grams of oleic acid per 100 c.c. of oil.

QUESTION.—How would you proceed in order to determine the per cent. by weight of oleic acid?

E. INSPECTION OF FLOUR.

1. Cold Water Extract.

Details of Procedure.—Weigh out on the flat scales 100 grams of flour. Transfer it to the graduated liter flask and thoroughly mix it with distilled water. Finally make up to the mark, stopper the flask, mix thoroughly by shaking frequently during one of the laboratory periods. Allow to stand over night.

Weigh the porcelain dish to the nearest milligram. Measure into the weighed dish by means of a pipette 50 c.c. of the cold water extract and evaporate to dryness on the steam bath. Dry, cool in a desiccator and weigh to the nearest milligram. Repeat the heating until two successive weighings show no alteration in weight.

Calculation of Result.—Per cent. cold water extract = residue from 50 c.c. of the filtrate multiplied by 20.

Note.—According to Wanklyn the cold water extract from a sound flour should not exceed 5 per cent.

The extract should not stand longer than 24 hours, as fermentation sets in and butyric acid is formed.

Question.—1. Of what value is this determination?

2. Determination of Gluten (Wiley).

Principle of the Method.—The flour is first made into a dough from which the starch and soluble material is washed out, leaving the gluten.

Apparatus.—Porcelain dish (8 cms.), aluminum dish, spatula, steam bath, balance and weights, desiccator.

Details of Procedure.—Weigh to the nearest centigram about 10 grams of flour. Transfer the weighed sample to a porcelain dish, add not more than 10 c.c. of water and then by use of a spatula work the mass up into a small ball, taking care not to allow any of the material to adhere to the sides of the dish. Allow the ball of dough to stand for one hour.

After standing knead the ball of dough in the hand under a small stream of water from the spigot until all the soluble

material and starch has been removed. Place the ball of gluten in cold water and allow to remain for one hour.

Accurately weigh an aluminum dish.

Remove the ball of gluten from the water, press it between the fingers until it is as dry as possible, then roll it into a small ball, place it in the weighed aluminum dish and weigh. Record the weight as moist gluten. Now place the dish and contents on the steam bath for twenty-four hours, then dry, cool in a desiccator, weigh and record the weight as dry gluten.

Calculation of the Result.—Per cent. gluten = 100 (weight of gluten)/(weight of sample).

Note.—Gluten is composed of two proteids, glutenin and gliadin. Wheat flour contains from 9 to 13 per cent. gluten.

3. Detection of Artificial Bleaching.

Note.—The artificial bleaching of flour consists in adding a small amount of nitrogen peroxide to the flour, which renders the yellow oil present nearly colorless.

(a) *Gasoline Method.*

Principle of the Method.—Gasoline dissolves the oil in the flour. The color of the oil is determined by observing the tint given to the solvent by the dissolved material.

Apparatus.—Glass-stoppered bottle (4 oz.).

Reagents.—Pure gasoline.

Details of Procedure.—Place about 20 grams of flour in the glass-stoppered bottle, then nearly fill it with gasoline. Shake well and allow the contents to settle. The gasoline from an unbleached flour so treated will have a distinct yellow color, while that from a bleached flour will be almost colorless.

Note.—Conduct a similar experiment on a flour known to be unbleached and compare the result.

(b) *Sulphanilic Acid—Alpha Napththylamine Method.*

Principle of the Method.—When sulphanilic acid $[C_6H_4 (NH_2) (HSO_3)]$ and α-naphthylamine hydrochloride $[C_{10} H_4 H_3 NH_2 HCl]$ react in the presence of nitrous acid we have formed the red compound, azo benzene-naphthylamine sulphonic acid $[C_6H_4 (HSO_3) N = NC_{10} H_4 H_2 NH_2]$. The reaction is of extreme delicacy.

Apparatus.—Glass-stoppered bottle (4 oz.).

Reagents.—1. Sulphanilic acid solution prepared by dissolving 0.5 grams of sulphanilic acid in 150 c.c. of dilute acetic acid (about 20 per cent.). (Keep well stoppered.)

2. Alpha naphthylamine hydrochloride solution made by dissolving 0.2 grams of alpha naphthylamine hydrochloride in 20 c.c. of glacial acetic acid and adding 130 c.c. of 20 per cent. acetic acid to it. (Keep well stoppered.)

3. Pure distilled water containing no nitrites.

Details of Procedure.—First make a preliminary test to determine the purity of the water to be used in the determination. Almost fill the glass-stoppered bottle with the water to be examined. Mix equal volumes of the solutions (1) sulphanilic acid and (2) alpha naphthylamine hydrochloride and then add 10 c.c. of the mixed reagent to the water. Stopper the bottle, shake well and allow to stand for twenty minutes. If at the end of that time the solution remains colorless or turns only a *very faint pink*, the water is suitable for this purpose.

Place a heaping teaspoonful of flour in the clean glass-stoppered bottle, nearly fill it with the pure water, add 10 c.c. of the mixed reagent, stopper, shake and allow to settle.

After twenty minutes observe.

A bleached flour will impart a tint to the solution varying from a pink to a deep red, depending upon the degree of the bleaching. Unbleached flour should give no more color than that imparted by the water itself.

Note.—The test should be accompanied by one made on flour known to be unbleached and the results compared.

Question.—What is the effect upon the finished loaf when bleached flour is used in the preparation?

4. Determination of Total Carbohydrates.

Principle of the Method.—Hydrolyze the carbohydrates with dilute acid whereby the starch is converted to sugar. Determine the quantity of sugar by Fehling's reagent.

Apparatus.—Erlenmeyer, condenser, 500 c.c. graduated flask, 25 c.c. pipette.

Reagents.—Hydrochloric acid (Sp. Gr. 1.125), Fehling's solution ("A" and "B").

Details of Procedure.—Accurately weigh about three grams of the sample. Transfer the same to an Erlenmeyer, add 200 c.c. of a mixture of 180 c.c. of water and 20 c.c. of hydrochloric acid (1.125), connect with a reflux condenser and boil for an hour and a half.

While this boiling is in progress, prepare a Gooch crucible, washing it successively with water, alcohol and ether, and then drying at 100 degrees, cooling in a desiccator and weighing.

After the carbohydrates have been refluxed for the time stated, cool, neutralize with Na(OH), transfer to the 500 c.c. graduated flask and make up to the mark with distilled water. Mix well and filter a portion into a dry beaker.

Sixty c.c. of the mixed copper reagent are diluted with 60 c.c. of water and heated to boiling. Add 25 c.c. (pipette) of the dextrose solution and boil 2 minutes. Filter immediately through the Gooch. Wash successively with water, 10 c.c. of alcohol and 10 c.c. of ether. After the odor of ether has vanished dry at 100 degrees for one-half hour. Cool in a desiccator and weigh as Cu_2O.

Calculation of the Result.—"M" grams of Cu_2O = "P" grams of dextrose. (See tables U. S. Bulletin, No. 107, Dept. of Chemistry.)

Per cent. starch = 100 $(P \times 20 \times 0.9)$/(weight of sample).

5. Action of Heat on Starch.

Object of the Experiment.—The experiment is designed to show the physical and chemical reactions that take place in starch upon the application of heat.

Apparatus.—Test tubes, evaporating dish, Bunsen burner, oven, beakers, tripod.

Reagents.—Water, Fehling's solution, tincture of iodine or iodine dissolved in potassium iodide solution, nitric acid, starch.

Details of Procedure.—(a) To one gram of starch in a small beaker add 50 c.c. of cold water. Filter, and if necessary repeat several times until the filtrate is perfectly clear, and apply the test for starch in (B). What is the solubility of starch in cold water?

(b) Make a paste by rubbing a gram of starch with a very small quantity of water. Slowly add to the paste a small quantity at a time to 200 c.c. of boiling water and stir after each addition. Cool. (?). Test for starch by adding to a portion of the above clear liquid a few c.c. of tincture of iodine or of iodine dissolved in potassium iodide solution. A deep blue color indicates the presence of starch.

To another portion of the starch solution add mixed Fehling's solution and heat. (?). Consult an Instructor if necessary.

(c) Place about 20 grams of starch in a porcelain evaporating dish. Heat cautiously to a temperature of 210 to 280 degrees C. for one-half hour, stirring constantly. Cool. Place a pinch of the material in a test tube and try its solubility in cold water. (?). Add alcohol to the aqueous solution. (?).

Test a portion of this material for starch by the iodine test. (?). Has a chemical reaction taken place? How do you know? What is the new material? Give its chemical formula.

(d) Moisten ten grams of starch with dilute nitric acid. Dry the paste first on the steam bath and finally heat over a small flame from the Bunsen burner to a temperature of 110 degrees C. Test the material; (a) solubility, (b) iodine test, (c) Fehling's solution. What are your conclusions?

6. Action of Acids upon Starch.

Object of the Experiment.—Starch is treated with boiling acid and afterwards tested in order to determine whether a chemical reaction has taken place.

Apparatus.—Erlenmeyer (8 oz.), condenser, tripod, Bunsen burner, wire gauze, test tubes, beaker, microscope.

Reagents.—Hydrochloric acid (Sp. Gr. 1.125), Fehling's solution, tincture of iodine or iodine in potassium iodide; sodium hydroxide.

Details of Procedure.—Place about three grams of starch in an Erlenmeyer, add 200 c.c. of distilled water and 200 c.c. of the hydrochloric acid. Connect the Erlenmeyer with a reflux condenser and boil for half an hour.

Disconnect the apparatus, remove about 10 c.c. of the solution, cool under running water, neutralize with Na(OH), and apply the test for starch. If this test proves positive connect up

the apparatus and boil for another half hour. Repeat if necessary until iodine gives no indication of starch.

Neutralize a portion of the solution with Na(OH) and apply the Fehling's solution test. (?).

Evaporate a portion of the liquid on a large clock glass on the steam bath. Examine the residue under the microscope. Is it starch?

QUESTIONS.—1. What change has taken place in the starch?

2. Write the reaction for the change, remembering that it is a case of hydrolysis.

3. How could the principle of this experiment be applied for the quantitative determination of starch in some of the commercial material?

7. Conversion of Starch to Dextrose by Diastase.

PRINCIPLE OF THE METHOD.—The enzyme diastase found in malt extract converts starch into maltose and dextrin.

APPARATUS.—Beaker, test tube, thermometer Erlenmeyer, stirring rod.

REAGENTS.—Tincture of iodine or a solution of iodine in potassium iodide, Fehling's solution, malt extract or diastase prepared by one of the following methods:

(a) Digest pulverized malt for several hours with enough alcohol to cover it. Filter and set the solution aside until the alcohol evaporates, then dissolve the residue in water. This solution contains the diastase.

(b) Dry malted barley may be readily obtained from any brewery. Treat 15 to 20 grams of the freshly pulverized malt for several hours with 100 c.c. of water, shaking occasionally. Filter the solution and add two or three drops of chloroform to prevent the growth of fungi.

DETAILS OF PROCEDURE.—Treat three grams of starch in an Erlenmeyer with 50 c.c. of water. Immerse the flask in a beaker containing boiling water and stir the contents of the flask until they gelatinize. Cool to about 60 degrees C. and add 20 to 40 c.c. of the malt extract, and again immerse in a beaker containing water at 55 to 60 degrees. Test after a half hour for unconverted starch. (?). Repeat the digestion until all the starch has been converted to sugar. (?). Test with Fehling's solution.

F. BREAD.

1. Determination of Moisture.

Details of Procedure.—Weigh an aluminum dish to the nearest milligram. Add about 10 grams of the finely divided sample and weigh again. Heat in an air oven at 100 degrees for one hour and re-weigh. Repeat the heating for half an hour or until after two successive heatings there is no change in weight.

Calculation of Result.—Per cent. moisture = 100 (Loss of weight by volatilization)/(Original weight taken).

Note.—In the best samples of bread the moisture content varies from 33 to 40 per cent. A moisture content over 40 per cent. is considered objectionable.

Perform this experiment on a sample of fresh bread, then repeat it when the bread has become stale. Compare the results.

2. Acidity.

Apparatus.—Mortar and pestle, burette.

Reagents.—Phenolphthalein, N/10 Na(OH) or KOH.

Details of Procedure.—Weigh 10 grams of the sample to the nearest centigram. Transfer to a mortar and macerate with 100 to 150 c.c. of distilled water. Wash off the adhering particles from the pestle by a stream of water from the wash bottle. Add two or three drops of phenolphthalein and titrate with N/10 alkali.

Calculation of Result.—Calculate c.c. N/10 alkali per 100 grams bread, also per cent. lactic acid.

Note.—Procure a number of samples of bread varying in quality from excellent to bad and compare them as to acidity.

Bread is seldom if ever adulterated. Adulterants found in bread are generally due to the use of impure flour.

G. MOLASSES.

Note.—Before taking a sample of molasses for any determination, stir thoroughly, as sugar is apt to have crystallized out.

1. Determination of Reducing Sugar (Dextrose).

Principle of the Method.—The determination of sugar is

made by means of Fehling's solution, the reducing sugar being calculated to dextrose.

Reagents.—Fehling's solution (A and B).

Details of Procedure.—Weigh a 100 c.c. graduated flask. Place in it approximately 5 grams of molasses, weigh exactly and dilute slightly. If the solution is clear, continue the dilution to the mark; but if cloudy, add 2 to 5 c.c. of lead subacetate before diluting to the mark. Filter, if necessary, through a dry paper and take 25 c.c. of the clear liquid. Place in a 100 c.c. graduated flask. This flask now contains one-fourth of the original sample. In case lead acetate has been used, add sodium sulphate in sufficient amount to precipitate all the lead. Dilute to the mark, filter if necessary.

Place 30 c.c. of the "A" solution of Fehling's and 30 c.c. of "B" into a beaker containing 60 c.c. of distilled water and heat to boiling. Add 25 c.c. (pipette) of the prepared sample containing one-sixteenth of the original weight and boil for two minutes. Filter immediately through a Gooch previously washed with water, alcohol, ether, then dried, cooled in a desiccator and weighed. Wash the Cu_2O with hot distilled water, then 10 c.c. of alcohol, followed by 10 c.c. of ether. Dry in an air oven, cool in a desiccator and weigh.

Calculation of the Result.—From tables in the U. S. Bulletin, No. 107, Bureau of Chemistry, pages 50-51, obtain the weight of dextrose equivalent to the milligrams of Cu_2O in the determination, then

Per cent. dextrose = 100 (weight of dextrose
16)/(grams molasses).

2. Determination of Sucrose.

Principle of the Method.—The sucrose is inverted with hydrochloric acid and the total sugar then determined by Fehling's solution. The total dextrose thus found less the dextrose found in the preceding experiment is calculated to sucrose.

Reagents.—Fehling's solution ("A" and "B").

Details of Procedure.—Dilute an accurately weighed sample, approximately 1 gram to 100 c.c. with distilled water. Add 3 c.c. of concentrated HCl and heat to 70 degrees for ten minutes, then neutralize with Na OH and dilute to 500 c.c.

Place 30 c.c. each of "A" and "B" solutions of Fehling's in a beaker containing 35 c.c. of water. Heat to boiling, add from a pipette 50 c.c. of the sugar solution and boil for two minutes. From this point proceed as directed in the determination of dextrose.

H. 1. COLOR IN CANDY.

Procure some samples of highly colored candy. Dissolve in water, acidify with hydrochloric acid and boil with pieces of white woolen cloth, the latter having first been cleansed by washing in dilute alkali.

If the wool is dyed, boil it with weak ammonia till as much color is removed as possible. Acidify the solution and boil with a fresh piece of wool.

If this piece is dyed, we may infer the presence of coal tar dyes.

PREPARATION OF NORMAL SOLUTIONS.

A normal solution is one that contains one (1) gram-equivalent of the active radical per liter of solution.

Thus in the case of acids, hydrogen is the active radical, so that to make 1 liter of normal acid we require such a weight of the acid as contains one gram-equivalent of hydrogen radical. One gram-equivalent of hydrogen radical is contained in one mol of hydrochloric acid in one-half mol of sulphuric acid, etc.

In the case of bases, the active radical is (OH), so that to make 1 liter of a normal solution of a base we require one gram-equivalent of hydroxyl radical. One gram-equivalent of hydroxyl is contained in one mol of sodium hydroxide, in one-half mol of barium hydroxide, etc.

In the case of oxidizing solutions, available oxygen is the active radical. Thus 1 liter of a normal oxidizing solution will contain eight grams of available oxygen. Reducing solutions contain an amount of the solute capable of combining with eight grams of oxygen.

It is usually more convenient to prepare a solution of approximately normal concentration and then to determine the relation of its concentration to that of a normal solution by accurate analysis.

Normal Hydrochloric Acid.

Thus to prepare a solution of hydrochloric acid of approximately normal concentration and to determine its normality, proceed as follows:

Our concentrated hydrochloric acid has a specific gravity of 1.19 and contains 37.23 per cent. by weight of hydrogen chloride, therefore we have

$$1.19 \times V \times .3723 = 36.46$$
$$V = 82.3 \text{ c.c.}$$

Take 82 or 83 c.c. of the concentrated acid and dilute to one liter. Mix thoroughly and fill a burette with the solution. Take two portions, each accurately measured, of approximately 10 c.c. each. Dilute with water to about 200 c.c. and add an excess of a solution of silver nitrate and a few drops of nitric acid. Digest and stir in a subdued light till the liquid has become clear and bright. Allow to stand in the dark. Prepare a Gooch crucible, dry at 170 degrees C. and weigh. Filter, wash with 1 per cent. nitric acid, dry at 170 degrees C. and weigh.

Then we have: Normality $= 100 \times w/143.33$, where w is the grams of silver chloride obtained from 10 c.c. of the solution.

In preparing $^{n}/_{2}$ or $^{n}/_{10}$ acid proceed in the same way, starting with one-half or one-tenth of the quantity of concentrated hydrochloric acid.

Normal Sulphuric Acid.

Proceed in a similar manner to that employed in the case of hydrochloric, noting that concentrated sulphuric acid has a specific gravity of 1.84 and contains 95.6 per cent. H_2SO_4 by weight.

It is standardized by precipitating an accurately measured volume (approximately 10 c.c.) with barium chloride and weighing the barium sulphate so obtained in a Gooch crucible. Normality $= 100 \times w/233.4$, where w is the weight of barium sulphate obtained from 10 c.c. of the solution.

Normal Potassium Hydroxide.

Assume that pure stick potassium hydroxide of the laboratory contains 10 per cent. inert foreign matter, chiefly water. Take such a weight as will give 56 grams KOH, dissolve in water and dilute to 1 liter. Mix thoroughly.

Fill two burettes, one with an acid of known normality, the other with the solution just prepared. Take the initial readings and draw into a beaker or Erlenmeyer flask from 10 to 15 c.c. of one of the solutions, add one or two drops of methyl orange (or other suitable indicator) and run in the other solution till a permanent color change results. Then alternately add each of the solutions till the color change is produced by the least possible quantity (a fraction of a drop) of the more dilute solution. Avoid splashing and rinse the sides of the vessel before the final adjustment.

Calculate the normality of the solution from the mean of at least five trials.

CHOICE OF AN INDICATOR.

Methyl Orange.

Dissolve 0.022 grams in 100 c.c. water, add 0.67 c.c. $^n/_{10}$ HCl and filter after standing some time.

USES.—In titrating strong acids (HCl, HNO_3, H_2SO_4). H_3PO_4 reacts neutral when $Na H_2 PO_4$ has been formed; H_2SO_3 when $Na HSO_3$ has been formed. In titrating all inorganic bases with strong acids.

Lacmoid.

Dissolve 0.2 gram in 100 c.c. alcohol.

USES.—In titrating strong acids and bases; also ammonia.

Litmus.

Of such a concentration that three drops of the solution impart a distinct color to 50 c.c. water.

USES.—In titrating strong acids and bases and ammonia.

Phenol-Phthalein.

Dissolve 1 gram in 100 c.c. 86 per cent. alcohol.

USES.—In titrating acids and strong bases. Not for ammonia. $H_3 PO_4$ is neutral when $Na_2 HPO_4$ has been formed; $H_2 CO_3$ when $Na HCO_3$ has been formed.

This is the most sensitive indicator for the titration of acids.