LABORATORY MANUAL OF
AGRICULTURAL CHEMISTRY

HEDGES AND BRYANT

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LABORATORY MANUAL OF AGRICULTURAL CHEMISTRY

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PREFACE

This Laboratory Manual is the outgrowth of several years' experience in agricultural institutions in teaching chemistry in its various relations to agriculture. The directions are designed as a laboratory guide for students in agricultural chemistry. It is necessary that this course be preceded by a course in general or inorganic chemistry and accompanied by a course in the theory of agricultural chemistry. A course in quantitative analysis is not necessarily a prerequisite. At the beginning of the Manual, along with the preparation of standard solutions used later in the analysis of agricultural products, a few experiments in quantitative analysis have been given to illustrate the fundamental principles and more important methods of manipulation. Directions as to the setting up of apparatus, details of manipulation, use and care of the balance, chemistry, and stoichiometry should be given in lectures accompanying the laboratory practice. The authors did not consider it advisable to burden the laboratory guide with a large amount of explanatory notes giving reasons for each step in the directions. It was thought that this could be accomplished to better advantage by means of lectures accompanying the laboratory work, and by questions at the end of each experiment that are designed to encourage the student to think for himself and to do outside reading. This arrangement gives the teacher an opportunity to

present the theory in any particular manner he may desire and to come in closer contact with the students, thereby making the practice more interesting to them by the personal contact. The directions for each experiment are simplified, as far as it is considered advisable, in order that the student might be able to pursue the laboratory work with the least possible assistance from the teacher. In the Appendix is given a list of some of the most important works bearing on this subject so as to further the interest of the student by encouraging outside reading.

In preparing this Manual free use has been made of standard works on quantitative analysis, of the publications of the Association of Official Agricultural Chemists, and of the bulletins of the Bureau of Chemistry, United States Department of Agriculture. The authors desire to acknowledge their obligation to the "Letter-Files" and to the Yale University Press for the portrait of Professor S. W. Johnson used as the frontispiece.

COLLEGE STATION, TEXAS

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LABORATORY MANUAL OF
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PART I

PREPARATORY QUANTITATIVE ANALYSIS

INTRODUCTION

At the first laboratory period the student should check up the apparatus in the desk assigned to him and immediately report any shortage or broken apparatus to the instructor in charge. Each student should be required to turn in at the end of the term's work a desk fully equipped with a complete set of apparatus in perfect condition. He should also be required to make a wash bottle to be used in the analytical work, and have it inspected by the instructor. All records of the laboratory work should be recorded in a notebook in a neat and systematic manner.

Reports of experiments and answers to all questions should be submitted at the next period, after the experiment is completed. Cleanliness, neatness, patience, and the most careful attention to details of the directions cannot be overemphasized in laboratory manipulations.

Experiment No. 1

PREPARATION OF CLEANING MIXTURE

Dissolve 20 g. of commercial $K_2Cr_2O_7$ in 75 cc. of warm water, cool, and pour slowly into it, with constant stirring, 115 cc. of commercial sulphuric acid (H_2SO_4). The potassium dichromate solution should be placed in a No. 7 evaporating dish, if convenient, before pouring the sulphuric acid into it. When the solution is completely cooled, it should be transferred to a 250 cc. wide-mouthed bottle. (Be careful in making this mixture, so as not to spill it on your clothes.) Invert the burettes in the cleaning mixture, attach the rubber tubing, draw up to the 50 cc. mark, and allow to stand for at least half an hour before being used. (*See that the burette stopcocks work smoothly and are not clogged with grease.*) Coat the burette stopcocks with a very small amount of vaseline to prevent sticking. The cleaning mixture is to be used throughout the course. Avoid the addition of water to the cleaning mixture. A burette is clean when the drops of water will not adhere to the sides. Other useful cleaning mixtures are soap solutions, alcoholic KOH solutions, and ammoniacal alcohol solutions.

Chemistry

Both H_2CrO_4 and CrO_3 oxidize organic matter.

Questions

1. Why does cleaning mixture clean? 2. Write equation.
3. What would you use to clean grease from glassware? Give reason.
4. After using the mixture for some time, it may become green in color. Give reason.
5. What is meant by the terms *commercial* and *C. P. chemicals*?

Experiment No. 2

PREPARATION OF AN APPROXIMATE N/10 NaOH SOLUTION

A normal solution is one which contains the hydrogen equivalent of the active constituent in grams per liter; that is, the amount in a liter which brings into reaction 1.008 g. of hydrogen or 8 g. of oxygen, or their equivalent.

One molecule of NaOH brings into reaction one atom of hydrogen when it reacts with an acid. Therefore 40.058 g. of NaOH is equivalent to 1.008 g. of hydrogen; hence there are 40.058 g. of NaOH in one liter of normal NaOH solution, and 4.0058 g. in one liter of a N/10 NaOH solution.

Since the NaOH "sticks" contain about 10 to 20 per cent of water, weigh out (on rough balance) 5 g., dissolve in distilled water, and dilute to one liter. Shake the solution thoroughly.

Experiment No. 3

PREPARATION OF AN APPROXIMATE N/5 HCl SOLUTION

One molecule of hydrochloric acid (HCl) contains one replaceable hydrogen atom; therefore 36.458 g. of hydrogen chloride furnishes 1.008 g. of reacting hydrogen. In other words, the molecular weight of HCl (36.458) contains

1.008 parts of reacting or replaceable hydrogen. Therefore it requires 36.458 g. of hydrogen chloride (HCl) for one liter of a normal hydrochloric acid (HCl) solution, and 7.2916 g. for one liter of a N/5 hydrochloric acid (HCl) solution.

The specific gravity of the C. P. concentrated hydrochloric acid solution on the reagent shelf should be determined by means of a specific-gravity spindle or hydrometer.

EXAMPLE. Should the specific gravity of the C. P. concentrated hydrochloric acid solution on the reagent shelf be 1.175, it would contain 34.43 per cent of hydrogen chloride by weight (see table of specific gravities, p. 85). Therefore $1.175 \times .3443 =$ grams of hydrogen chloride in 1 cc. of the C. P. concentrated hydrochloric acid solution, and $\frac{7.2916}{1.175 \times .3443} = 18$ cc. of C. P. concentrated hydrochloric acid required to furnish approximately 7.2916 g. of hydrogen chloride. This is the weight or amount of hydrogen chloride required for one liter of a N/5 hydrochloric acid solution, providing the concentrated acid is of the above specific gravity.

Measure out the amount of the concentrated C. P. hydrochloric acid required by your calculations (in the above example it would be 18 cc.) and dilute to one liter with distilled water. Shake the solution thoroughly before using.

Experiment No. 4

DETERMINATION OF EQUIVALENT VOLUMES

Rinse out two burettes (which have been standing in cleaning mixture) with distilled water. After each burette is rinsed with distilled water, it should be rinsed again with a 5 cc. portion of the solution to be used in the respective burette, so as to remove any adhering particles

of water (letting the solution run through the tip each time). Label the burette according to the solution to be used therein. Fill one of the burettes* a little above the zero mark with the approximate $N/10$ NaOH solution. Open the stopcock or pinchcock cautiously until the reading of solution in the burette is zero. In the same manner fill the other burette with the approximate $N/5$ HCl solution, using the same care as to washing, etc. Draw out approximately 10 cc. of the NaOH solution into a 250 cc. Erlenmeyer flask or beaker. Dilute to 50 cc. with distilled water, add one drop of methyl orange, and then rapidly add the HCl solution until the solution in the flask or beaker changes color, indicating an acid solution. Now bring the flask or beaker under the burette containing the NaOH, and add the NaOH solution more slowly, until the color turns yellow. Then bring the flask or beaker under the burette containing the HCl, and add the HCl solution slowly until the solution turns red. Continue this operation until a point is obtained where one drop, or even half a drop, of either solution will cause a color change. When this point — known as the *end-point* — is reached, allow the solutions in the burettes to drain for a minute, and then read the exact volume of each solution used.

Record in your laboratory notebook the exact volume of solution used. As a check on this determination and also as work with indicators, make titrations, using phenolphthalein, cochineal, and methyl red as indicators. Make at least two titrations with each indicator. Different determinations should agree within .05 cc. Take the average of the titration for the calculations. From the data

* It is advisable to use a rubber-tipped burette for the alkali solution.

obtained calculate and report the cubic centimeters of HCl solution neutralized by 1 cc. of the NaOH solution. Also calculate and report the cubic centimeters of NaOH solution neutralized by 1 cc. of the HCl solution.

Questions

1. How many grams of the following reagents will be required for one liter of a normal solution; one liter of fifth-normal solution: HNO_3 , H_2SO_4 , H_3PO_4 , $\text{H}_2\text{C}_2\text{O}_4$, $2\text{H}_2\text{O}$, NH_4OH , NaHCO_3 , AgNO_3 , K_2SO_4 ? 2. How many cubic centimeters (cc.) of N/10 HCl = 15 cc. of N/1 NH_4OH ? N/5 K_2CO_3 ? N/10 NaOH? N/1 KOH = 10 cc.? N/4 $\text{H}_2\text{C}_2\text{O}_4$? N/2 H_2SO_4 ? 3. If 8 g. of a sample containing some NaOH were dissolved in 200 cc. of distilled water, and 20 cc. of this solution were equivalent to 15 cc. of a N/5 acid, what would be the percentage of NaOH in the original material?

Experiment No. 5

I. PREPARATION OF A PERFORATED OR GOOCH CRUCIBLE

Prepare two crucibles in the following manner: Place the Gooch funnel in the neck of a filter flask by means of the rubber stopper, stretch the rubber band over the funnel, and place the crucible in the opening. Connect the filter flask, by means of a rubber tubing, to the filter pump, and before turning on the suction pour into the crucible some of the coarse, suspended asbestos* (obtained by shaking the bottle). When the water has filtered through, turn on the suction and pour on some of the finer asbestos (supernatant liquid obtained on allowing the solution to settle a short time). Tap well with flattened glass rod, and add another layer. Then wash

* Prepared according to directions on page 88.

with at least 100 cc. of distilled water, and dry in the air bath at 130° – 150° C. for two hours. Cool in *desiccator*, and weigh accurately to tenths of a milligram. (Crucibles

ANALYTICAL BALANCE

should be handled only with clean forceps.) The asbestos film should be thin enough to be translucent when wet, but not so thin that it will be liable to permit a fine precipitate to pass through.

Do not put any labels on the crucibles, and do not mark them. They may be identified by placing them in a labeled funnel.* The crucibles should be treated in the same way before and after receiving the precipitates. This applies to the length of time in the drying oven, the heat applied, and the length of time they stand in the desiccator before weighing.

Questions

1. What is asbestos, and how is it obtained? 2. How should asbestos be prepared for use in a Gooch crucible? 3. In Experiment No. 5, why not filter through a filter paper? 4. Give three precautions that should be taken when using a desiccator. 5. Name four reagents that could be used in a desiccator, for drying purposes, and discuss their efficiency.

II. STANDARDIZATION OF HCl SOLUTION BY GRAVIMETRIC METHOD

Into two 250 cc. Erlenmeyer flasks, which have been cleaned with cleaning mixture and carefully rinsed with distilled water, measure out accurately from a burette 25 cc. portions of HCl solution. (Allow the burette to drain for about five minutes before reading.) Add 75 cc. of distilled water, 5 cc. of C. P. concentrated HNO_3 solution, and then a solution of silver nitrate† (AgNO_3) very gradually and with constant agitation of solution until the precipitation is complete. Close the flask with a clean rubber stopper, wrap in a black cloth, and shake the flask vigorously for several minutes until the AgCl

* Crucibles may be permanently marked for identification either with China paints by Yoder's method (circular of Bureau of Chemistry) or, more conveniently, by burning in a "grease" Prussian blue pencil mark. J. E. Huber, *Chemist Analyst*, January, 1915, p. 25.

† Prepared according to directions on page 89.

is flocculated and the supernatant liquid is perfectly clear. When the precipitation is complete, the supernatant liquid quickly becomes clear. To this clear portion a drop of silver nitrate may be added to decide whether enough has been added for complete precipitation of the hydrochloric acid. Avoid the addition of an excess of the silver solution. It is necessary to protect the flask containing the precipitate from the light, as much as possible, as light tends to decompose the silver chloride precipitate (AgCl).

Let the precipitate settle, then filter off the supernatant liquid through the *tared* Gooch crucible by suction, introducing as little of the precipitate as possible on the filter. Wash with about 150 cc. of a cold 1 per cent HNO_3 solution by similar decantation through the Gooch. Transfer the precipitate, without loss, from the flask to the tared crucible, with about 100 cc. more of a 1 per cent HNO_3 solution, making sure that none is left in the flask or on the rubber stopper, and wash thoroughly with a 1 per cent HNO_3 solution until 15 cc. of the filtrate does not give a test for silver nitrate (AgNO_3). Suck fairly dry. Dry the crucible and contents for two hours at 130° – 150° C., cool in desiccator, and weigh. Heat again for one hour, cool, and reweigh. Repeat until weight is constant. Duplicates must not differ in weight more than 1 mg. At least four trials should be made.

Chemistry

Mol. wt. of AgCl : mol. wt. of HCl :: weight of ppt. : x
 x = grams of hydrogen chloride (HCl) in number of cubic centimeters of hydrochloric acid solution used.

$\frac{x}{\text{Volume of HCl used}} = \text{grams of hydrogen chloride (HCl) in 1 cc. of the hydrochloric acid solution, which is known as the titre of that solution.}$

Questions

1. Show how to make a N/10 HCl solution, a N/5 H₂SO₄ solution, a N/10 HNO₃ solution, a N/20 NaOH solution, using HCl, sp. gr. 1.185; H₂SO₄, sp. gr. 1.8; HNO₃, sp. gr. 1.4; NaOH, containing 20 per cent of water. 2. What is the percentage of Ag in AgCl? Fe in FeSO₄(NH₄)₂SO₄ · 6 H₂O? S in BaSO₄? and CaO in CaCO₃? 3. How many cubic centimeters of HCl (sp. gr. 1.2) are necessary to precipitate completely the silver in 2 g. of AgNO₃? 4. How many cubic centimeters of N/10 AgNO₃ solution will be necessary to precipitate the chlorine from .12 g. of CaCl₂? 5. What effect has light upon AgCl? Explain.

Experiment No. 6

I. STANDARDIZATION OF THE HCl SOLUTION BY VOLUMETRIC METHOD

Weigh out accurately 0.2 to 0.3 g. portions of C. P. Na₂CO₃ into 250 cc. Erlenmeyer flasks or beakers, and dissolve each portion in 50 cc. of distilled water. Add one drop of methyl orange to one portion, and titrate with HCl solution until the first tinge of pink appears. Repeat the operation with the second portion. Record accurately the burette readings.

Chemistry and Calculations

Mol. wt. of Na₂CO₃ : mol. wt. of 2 HCl :: wt. of Na₂CO₃ : x
 106.1 : 72.916 :: weight of Na₂CO₃ taken : x

$\frac{x}{\text{Volume of HCl used}} = \text{grams of hydrogen chloride (HCl) in 1 cc. of the hydrochloric acid solution, which is known as the titre of that solution. If the duplicates}$

APPARATUS FOR QUANTITATIVE ANALYSIS

1, Erlenmeyer flask (500 cc.); 2, wash bottle; 3, measuring flasks (500 and 1000 cc.); 4, burettes and burette holder; 5, desiccator; 6, graduated cylinder (50 cc.); 7, indicator bottles; 8, specific-gravity hydrometer and cylinder; 9, porcelain evaporating dish; 10, porcelain crucible and cover; 11, pipette (25 cc.)

do not agree to within 0.05 mg., the work must be repeated until the duplicates do agree. The results of this experiment should check with those obtained in Experiment No. 5. Report the titre of the HCl solution as obtained in the two experiments.

II. CALCULATION OF THE TITRE OF THE NaOH SOLUTION

By the use of the titre of the hydrochloric acid solution determined in Experiment No. 5 and the volume of hydrochloric acid solution neutralized by 1 cc. of the NaOH solution, as determined from the equivalent volume work (Exp. No. 4), calculate the titre of the NaOH solution.

Chemistry

$40.008 : 36.458 :: x : (\text{titre of HCl solution})$ times (the cubic centimeters of HCl neutralized by 1 cc. of the NaOH).

x = grams of NaOH in 1 cc. of the NaOH solution, which is known as the titre of that solution.

Questions

1. Define a normal solution, a standard solution, and a per cent solution. 2. Give difference between N/1 HCl and 1 per cent HCl solution. 3. Give the titre of the following solutions: 1.25 per cent NaOH; 1.25 per cent H_2SO_4 ; 4 per cent NH_4OH . 4. What is the difference, if any, between a 5 per cent NaOH and a N/5 NaOH solution?

Experiment No. 7

DETERMINATION OF THE STRENGTH OF AN UNKNOWN ALKALI SOLUTION

After cleaning a burette and rinsing with a 5 cc. portion of the unknown solution, fill it to the zero mark, according to directions in Experiment No. 4. In the same manner fill the other burette with the N/5 HCl solution. Titrate the one solution against the other, using methyl orange as an indicator and the same volumes as in the

equivalent volume work (10 cc. of the solution diluted to 50 cc., etc.). Run duplicate determinations. Calculate and report grams of NaOH per cubic centimeter of the unknown solution.

Experiment No. 8

DETERMINATION OF THE STRENGTH OF AN UNKNOWN ACID SOLUTION

Perform as in preceding experiment, using your standard N/10 NaOH solution. Run duplicate determinations. Calculate and report grams of HCl per cubic centimeter of the unknown solution.

Experiment No. 9

DETERMINATION OF THE STRENGTH OF AN UNKNOWN K₂CO₃ SOLUTION, USING METHYL ORANGE AS INDICATOR

Measure out, with burette, two 20 cc. portions of the unknown K₂CO₃ solution, dilute with 50 cc. of distilled water, and titrate in the same manner as with Na₂CO₃ in Experiment No. 6. Make at least two trials, and more if necessary to make titrations check. Calculate and report the amount of K₂CO₃ and also its equivalent in K₂O per cubic centimeter of the unknown solution.

Experiment No. 10

DETERMINATION OF THE STRENGTH OF AN UNKNOWN K₂CO₃ SOLUTION, USING PHENOLPHTHALEIN AS INDICATOR

Measure out, with burette, two 20 cc. portions of the unknown K₂CO₃ solution, dilute with 50 cc. of distilled water, and titrate in the same manner as in Experiment

No. 9, using phenolphthalein as indicator. Calculate and report the amount of K_2CO_3 and its equivalent in K_2O per cubic centimeter of the unknown solution. The phenolphthalein indicates only half the carbonates, as indicated by the equation

Experiment No. 11

DETERMINATION OF THE STRENGTH OF AN UNKNOWN NaOH AND Na_2CO_3 SOLUTION

In the preceding experiments it was shown that the hydroxides of sodium and potassium acted differently from the carbonates toward some indicators and that the carbonates acted differently with different indicators. When phenolphthalein is used as indicator, all the metal of the hydroxide is neutralized, while only half is neutralized in the carbonate. When methyl orange is used as indicator, all the metal of hydroxide and carbonate is neutralized. It ought, therefore, to be possible to tell the amount of a carbonate in a solution of a hydroxide by making titrations, using methyl orange and phenolphthalein as indicators.

Measure out, with burette, 10 cc. of the unknown solution; dilute with 50 cc. of distilled water; and titrate, using phenolphthalein as indicator,* noting the cubic centimeters of acid required. Measure out another portion of 10 cc., and titrate, using methyl orange as indicator. The phenolphthalein shows all the hydroxide and half the carbonate; the methyl orange shows all the hydroxide and all the carbonates. From data obtained calculate and report

* Prepared according to directions on page 88.

the amounts of carbonate and hydroxide per cubic centimeter of the unknown solution. Write all equations illustrating the reaction and give methods of calculating results.

Question

Discuss the use of indicators.

References

COHN. Indicators and Test Papers.

MORSE. Exercises in Quantitative Analysis, pp. 110-115.

NOYES, W. A. A Textbook of Chemistry, pp. 387-390.

STIEGLITZ. *Journal of American Chemical Society*, Vol. XXV, p. 1114.

SUTTON. Volumetric Analysis.

TIZARD. Indicators.

TREADWELL-HALL. Analytical Chemistry, Vol. II, pp. 426-435.

Experiment No. 12

DETERMINATION OF ACETIC ACID IN VINEGAR

The formula of acetic acid is $\text{HC}_2\text{H}_3\text{O}_2$. It has one replaceable hydrogen atom. Take 5 cc. of vinegar, measured by means of a burette or pipette, dilute to 100 cc. with distilled water, and titrate with your standard N/10 NaOH solution. Run duplicates. Use phenolphthalein as the indicator in the titration. Calculate and report grams of acetic acid in 1 cc. of the vinegar.

Questions

1. Considering that 1 cc. of vinegar weighs 1 g., what is the per cent of acetic acid in the sample of vinegar examined?
2. What is the standard in your state and in the United States for pure vinegar as to acidity and total solids?

Reference

LEACH. Food Inspection and Analysis (3d ed.), p. 772. 1914.

Experiment No. 13

DETERMINATION OF TOTAL SOLIDS IN VINEGAR

Weigh a small porcelain evaporating dish which has been previously cleaned and dried. Measure out exactly, with a burette, 10 cc. of the sample of vinegar to be analyzed, and evaporate this to dryness on a water bath; then dry for three hours in a water oven (or drying oven) at the temperature of boiling water. Cool the dish and solids in desiccator, and weigh. From the figures obtained calculate and report the percentage of total solids in the sample. Make two determinations.

Question

What is indicated by a low total-solids content? Discuss.

Experiment No. 14

DETERMINATION OF ASH IN VINEGAR

Take the solid residues from the previous experiment and ignite over a low flame until you obtain a grayish-white ash. Cool the dish and ash in desiccator, and weigh. The weight obtained minus the weight of the dish represents the weight of the ash in the amount of vinegar used. From the results obtained calculate and report the percentage of ash in the sample. Make two determinations. Test for water-soluble phosphates. Also test for phosphates insoluble in water but soluble in dilute hydrochloric acid.

An Argand burner with mica chimney or an electric muffle furnace may be successfully used for ashing. The

flame is kept low until charring is complete, then turned as high as it can be raised without smoking.

Questions

1. Explain the relationship of phosphates to the purity of vinegar. 2. Give a quick method for the manufacture of vinegar. 3. Explain the conditions necessary for the making and keeping of apple vinegar. 4. Compare the composition of cider with that of cider vinegar.

Reference

LEACH. Food Inspection and Analysis (3d ed.), pp. 759-782. 1914.

Experiment No. 15

PREPARATION OF STANDARD POTASSIUM PERMANGANATE SOLUTION (KMnO_4)

Dissolve 3.25 g. of KMnO_4 in 1 liter of distilled water. Let the solution stand until the next laboratory period, and then filter through a properly prepared perforated or Gooch crucible (prepared as in Exp. No. 5, Part I). Standardize the KMnO_4 solution against a known solution of ferrous ammonium sulphate ($\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6 \text{H}_2\text{O}$) as follows:

Place 3.5 g. (exactly weighed) of C. P. ferrous ammonium sulphate in a 200 or 250 cc. measuring flask, and make up to the mark with distilled water. Shake until the salt is completely dissolved. By means of a pipette or burette measure out 50 cc. of the ferrous ammonium sulphate solution into a beaker, add 25 cc. of sulphuric acid (H_2SO_4) strength 1:4, and dilute to about 100 cc. with distilled water. Add the KMnO_4 solution from a burette until one drop gives a permanent pink color. At least three titrations should be made. From the data obtained and

the following equation calculate the amount of iron (Fe) oxidized by 1 cc. of the KMnO_4 solution.

Observe that one seventh of the weight of the ferrous ammonium sulphate is iron (Fe).

Questions

1. Show that one seventh of the weight of ferrous ammonium sulphate is iron. 2. How many grams of KMnO_4 would be required for a N/10 KMnO_4 solution? How many grams of $\text{K}_2\text{Cr}_2\text{O}_7$ would be required for a N/10 $\text{K}_2\text{Cr}_2\text{O}_7$ solution? 3. Problem: weight of iron ore taken is .2 g.; volume of N/10 $\text{K}_2\text{Cr}_2\text{O}_7$ required for titration is 20 cc. What is the percentage of Fe in the sample?

Experiment No. 16

DETERMINATION OF THE AMOUNT OF CALCIUM OXIDE IN LIMESTONE

The sample of lime, limestone, or material containing calcium ground to a fine powder is thoroughly mixed, and approximately 2 g. of the mixture (exactly weighed) is placed in a beaker with 25 cc. of concentrated hydrochloric acid (HCl) and 25 cc. of distilled water. If necessary, the solution is heated to get the calcium compounds in solution. Possibly all the material does not go into solution, as the material may contain some silica (SiO_2). The calcium salts are with a little heating readily dissolved in the dilute acid. After the material is in solution, the solution in the beaker is transferred by filtering into

a 200 or 250 cc. measuring flask. Distilled water is used to wash out the beaker and to wash any material in the filter free of any soluble substance. The solution in the measuring flask is then made up to the mark with distilled water, shaken until thoroughly mixed, and two portions of 25 cc. each are measured out by means of a pipette or burette and placed in two beakers. To each of the portions 25 cc. of distilled water is added, then ammonium hydroxide (NH_4OH) solution is added until alkaline. The solution is heated to boiling, and while boiling a solution of ammonium oxalate* ($(\text{NH}_4)_2\text{C}_2\text{O}_4$) is added dropwise until the calcium is completely precipitated as calcium oxalate. The complete precipitation of the calcium is determined by allowing the precipitate to settle, and adding a drop of the ammonium oxalate solution to the supernatant liquid. If the precipitation is not complete, a precipitate will be formed; in which case the solution is to be treated again as at first.

Allow the precipitate to settle, filter while hot, and wash free from soluble oxalates, using hot water. The precipitate in the beaker should be thoroughly washed free of soluble oxalates, but it does not need to be completely transferred to the filter paper, as the filter paper and precipitates are later to be returned to the same beaker in which the precipitation took place. After the precipitate in the beaker and in the filter paper is thoroughly washed, return the filter paper and precipitate to the beaker from which it was filtered, add about 50 cc. of water and 10 cc. of sulphuric acid (1:1), heat nearly to boiling, and titrate with the standard KMnO_4 solution until you get a permanent pink color.

* Prepared according to directions on page 89.

Precaution. Do not heat the solution to boiling, but bring it nearly to boiling. Explain. Save standard KMnO_4 solution for other experiments.

Calculation of Results

Write all equations for the solution of the calcium compound and its precipitation. The reaction with KMnO_4 gives the following equation:

By comparing the equations for oxidation of Fe and CaC_2O_4 , it will be seen that the same amount of KMnO_4 oxidizes twice as much of the iron compound as of the calcium oxalate. As the molecular weight of CaO and the atomic weight of Fe are about the same, the factor for iron divided by 2 gives the factor for CaO; or the titre of KMnO_4 in terms of Fe divided by 2 gives the titre of KMnO_4 in terms of CaO. Calculate and report from the results the percentage of CaO in the sample.

Questions

1. If you had a sample of pure limestone, what per cent of CaO should you obtain by its analysis? 2. How many pounds of CaO and Ca(OH)_2 are equivalent to 100 lb. of air slaked lime? 3. One cubic centimeter of N/10 KMnO_4 solution will oxidize .0056 g. of iron (Fe). If .2 g. of a sample of barium oxalate requires 10 cc. of the KMnO_4 solution, what is the percentage of barium oxalate in the sample?

PART II

ANALYSIS OF FEEDSTUFFS

PREPARATION OF A SAMPLE OF FEEDSTUFF FOR ANALYSIS

The sample of feedstuff should be prepared for analysis by grinding the material to pass through a sieve having circular perforations 1 mm. in diameter. After sample is prepared and thoroughly mixed, it should be kept in a well-stoppered bottle. Enough of the sample should be prepared for the complete analysis.

Experiment No. 17

DETERMINATION OF MOISTURE IN FEEDSTUFFS

Weigh out exactly 2 g. of the feed sample in a weighed watch glass, and dry for three hours in a water oven at the temperature of boiling water; cool in desiccator, and weigh rapidly. Heat again, and weigh at intervals of one-half hour until the material ceases to lose weight. Make duplicate determinations. Calculate and report the percentage of moisture in the sample.

Question

Why heat at the temperature of boiling water, as specified, instead of at the temperature of 100° C. ?

Experiment No. 18

DETERMINATION OF ASH IN FEEDSTUFFS

The residue from Experiment No. 17, or a weighed amount (from 2 to 3 g.) of the original material, is ignited gently over a small flame in a weighed porcelain crucible to

ELECTRIC DRYING OVEN

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a constant weight. Be sure to weigh the crucible before placing the weighed sample of feed in it. Make duplicate determinations. Calculate and report the percentage

of ash in the sample examined. Make a qualitative analysis of plant ash for essential elements of plant food, and report results.

Question

What value has the ash determination in the analysis of feedstuff ?

Experiment No. 19

DETERMINATION OF CRUDE PROTEIN IN FEEDSTUFFS

Apparatus

Kjeldahl flasks of 800 cc. for both digestion and distillation, a safety bulb, and a condenser.

Weigh out accurately a convenient quantity of the feed sample (from 0.7 to 1.4 g.) and transfer it to the Kjeldahl flask. The neck of the flask should be dry, so that none of the material sticks on the neck. Add 25 cc. of concentrated C. P. H_2SO_4 and approximately 0.3 g. of CuSO_4 . Support the flask in a sloping position on a wire gauze or on a piece of asbestos board with a hole in the center. Heat gradually to boiling with a Bunsen flame (heating should be done in a hood or some arrangement made to remove the fumes so they will not get into the room).* As soon as the acid is boiling freely and any frothing has ceased, remove the flame; and when the contents have cooled, add about 5 g. of C. P. K_2SO_4 . The heating is renewed and continued until the material is completely oxidized. This will be shown by the acid's becoming clear and having only a slight blue or green tint. After removing the flame a few small pieces of

* Fumes can be removed by means of an earthenware or lead fume duct or by means of a Sy fumeless digestion apparatus.

KMnO_4 may be added to make sure that the oxidation is complete. This should be added until it permanently colors the solution. The flask and contents are allowed to cool completely, then 200 to 250 cc. of distilled water is added. Through a thistle tube which should reach to the bottom of the flask, or by slanting the flask and pouring down the side, there is now added about 75 cc. of

KJELDAHL DIGESTION APPARATUS

concentrated NaOH solution* (free of carbonates). Do not shake the solution in the flask before it is connected with the safety bulb and distillation apparatus. A small piece of granulated zinc or a little zinc dust is added to prevent bumping in the subsequent distillation. Insert the safety bulb in the flask and connect with a condenser. Gently shake the flask to mix its contents, and distill the ammonia into a measured amount (25 cc.) of the standard

* Prepared according to directions on page 89.

N/5 HCl solution (acid must be measured exactly from a burette). The receiver may conveniently be a 250 cc. Erlenmeyer flask. The end of the distillation tube should extend down into the standard HCl solution. When about 100 cc. have been distilled over, making a volume of approximately 125 cc. in the flask, the distillation may be stopped.

KJELDAHL DISTILLATION APPARATUS (STEAM)

Caution. Before removing the flame from under the distilling flask or cutting off the steam (if steam is used for the distillation) remove the receiving flask so as to keep the liquid from being sucked back into the distilling flask when the flame is removed. Titrate the distillate with your standard N/10 NaOH solution, and from the data obtained, calculate the percentage of nitrogen in the feedstuff. The indicator, which should be *cochineal* or *methyl red*, may be placed in the receiver with the acid

at the beginning of the distillation. Should the indicator show an alkaline reaction during the distillation, it means that not enough acid has been used to neutralize all the ammonia distilled. Multiply the percentage of nitrogen by 6.25 to obtain the percentage of crude protein in the sample. Run duplicate determinations. The duplicate nitrogen determinations should check within .02 per cent.

NOTE. Accurate work requires that a blank digestion and distillation be made with some substance that is nitrogen free, in order to test the purity of the reagents.

Questions

1. Give reasons for the use of each substance in the determination of nitrogen. 2. Why multiply the percentage of nitrogen by 6.25 to obtain the percentage of crude protein? Discuss.

Experiment No. 20

DETERMINATION OF ETHER EXTRACT (FAT) IN FEEDSTUFFS

Apparatus

One extraction thimble and extraction apparatus.

Place 2 or 3 g. (weighed exactly) of the sample of feedstuff in an extraction thimble, and a small piece of cotton in the opening of the thimble to hold the feedstuff in and also to distribute the ether during the extraction. Dry the thimble containing the feedstuff in the oven at the temperature of boiling water for two hours, to remove the moisture, and then extract with anhydrous, alcohol-free ether* for twelve hours. Use apparatus for the extraction as illustrated in the lectures or specified by the instructor. The receiving flask for the fat should be cleaned, dried, and weighed before the extraction is begun.

* Prepared according to directions on page 89.

After the extraction is complete, evaporate off the ether on a water bath from the receiving flask (recovering the ether, if possible), and dry the extract in the weighed vessel at

EXTRACTION APPARATUS FOR DETERMINATION OF FAT
(ETHER EXTRACT)

the temperature of boiling water for one-half hour; cool in desiccator and weigh. Repeat the drying and weighing at half-hour intervals until a constant minimum weight is obtained. The increased weight of the flask over the original weight of the flask when clean, empty, and dry is due to the weight of the fat extracted.

EXAMPLE

Let x = amount of feedstuff used.

Let y = weight of flask plus weight of fat.

Let z = weight of flask when empty, dry, and clean.

Then $y - z$ = weight of fat in x g. of feedstuff.

$$\frac{y - z}{x} \times 100 = \text{percentage of ether extract (fat) in the feedstuff.}$$

Make duplicate determinations. Calculate and report the percentage of ether extract in the sample of feedstuff.

NOTE. The term *ether extract* is used to represent the materials that are soluble in ether. Besides fats and oils the ether extract generally contains some waxes, resins, chlorophyll, coloring matters, and, in some cases, phosphorized fats and fatlike bodies containing both nitrogen and phosphorus.

Questions

1. What is ether? 2. How is it made? 3. What are some of its properties? 4. What impurities would most likely be found in ether? 5. How can these impurities be most conveniently eliminated? 6. What are sulphuric ether and petroleum ether? 7. Name the other solvents which may be used for the extraction of fat, besides ether, and give the temperature at which each boils. 8. Why is the heating value of a fat higher than that of a carbohydrate? 9. Why is the feedstuff dried before the extraction of fat, and why is anhydrous, alcohol-free ether used?

Experiment No. 21

PREPARATION OF A 1.25 PER CENT H_2SO_4 SOLUTION AND
A 1.25 PER CENT NaOH SOLUTION

A 1.25 per cent sulphuric acid solution contains 12.5 g. H_2SO_4 per liter. Determine the specific gravity of the C. P. concentrated sulphuric acid, and calculate the number of cubic centimeters of this solution that will contain 12.5 g. of H_2SO_4 (see Appendix, p. 85). Measure out the

calculated amount in a graduated cylinder, and dilute to one liter with distilled water. Titrate this solution against your standard alkali, and adjust the strength, if necessary, until the solution contains 0.0125 g. of H_2SO_4 per cubic centimeter.

Weigh out the amount of NaOH (sticks, free of carbonates) required for a liter of a 1.25 per cent NaOH solution (the sticks of NaOH generally contain from 10 to 20 per cent water). Shake thoroughly until the NaOH is dissolved. Titrate this solution against the 1.25 per cent H_2SO_4 solution, and adjust the strength until correct.

Questions

1. 10 cc. of 1.25 per cent H_2SO_4 = (?) cc. N/10 NaOH?
2. 10 cc. of 1.25 per cent NaOH = (?) cc. N/5 HCl?
3. 10 cc. of 1.25 per cent H_2SO_4 = (?) cc. 1.25 per cent NaOH?

Experiment No. 22

DETERMINATION OF CRUDE FIBER IN FEEDSTUFFS

Apparatus

One 500 cc. Erlenmeyer flask; one condenser; two Gooch crucibles; 1.25 per cent H_2SO_4 solution; 1.25 per cent NaOH solution.

Place the residue from Experiment No. 20 in a 500 cc. Erlenmeyer flask. Add 200 cc. of boiling 1.25 per cent H_2SO_4 solution, connect with a reflux condenser, and boil gently for thirty minutes. Filter through a Gooch crucible containing a small quantity of asbestos, or a Büchner funnel, using closely woven linen as a filter. Wash with boiling water until the washings are free from acid. Rinse the substance remaining in the crucible or on the linen back into the Erlenmeyer flask with 200 cc. of a boiling

solution of 1.25 per cent NaOH (free from carbonates). Boil at once for thirty minutes as before. As soon as the boiling is completed, filter through a Gooch crucible or Büchner funnel, as before, wash several times with boiling water, once with dilute HCl (1 : 1), and then with boiling water until the washings are free from acid and are neutral. If linen is used as the filter, the material remaining on the linen has to be transferred to a porcelain crucible before drying. Dry the porcelain crucible or Gooch crucible containing the crude fiber, asbestos, and mineral matter in an oven at the temperature of 110° C. until completely dry, and weigh to constant weight. Then ignite the material in the porcelain crucible or Gooch crucible with a Bunsen flame, cool the crucible in a desiccator, and weigh to constant weight. The loss in weight represents the weight of the crude fiber in the sample of feedstuff examined. Make duplicate determinations. Calculate and report the percentage of crude fiber in the sample of feedstuff.

Questions

1. What is crude fiber? 2. Is it a compound or a mixture? 3. What substances are removed by each digestion? 4. Why boil for a specified time? 5. Why are the solutions of acid and alkali of this particular strength used, and why are they used in a certain order? 6. Discuss the value of the crude-fiber determination in the consideration of the analysis of a feedstuff.

Experiment No. 23

DETERMINATION OF NITROGEN-FREE EXTRACT IN FEEDSTUFF

Nitrogen-free extract is usually obtained by difference. Subtract from 100 per cent the sum of the percentage of moisture, ash, ether extract (fat), crude fiber, and crude

protein. The result is the percentage of nitrogen-free extract in the sample.

Report the results in the following form:

DETERMINATION No. 1	DETERMINATION No. 2
Moisture.....	
Ash.....	
Ether extract.....	
Crude fiber.....	
Crude protein.....	
Total x	Total x'
$100 - x =$ percentage of nitrogen-free extract.	

Questions

1. What substances constitute the nitrogen-free extract?
2. Make a comparison of the analyses of two samples of feed-stuff of the same kind, and give reasons for conclusions in regard to their feeding value.
3. A sample of feedstuff weighing 200 g. after being air-dried weighed 50 g. The analysis of the air-dried sample was as follows:

Moisture	4.40%
Crude protein	10.75%
Ether extract (fat)	1.15%
Nitrogen-free extract	64.86%
Crude fiber	12.82%
Ash	6.02%

Report the analysis on the basis of the original material and also on the moisture-free basis.

PART III

CHEMICAL ANALYSIS OF SOIL

DIRECTIONS FOR TAKING A SAMPLE OF SOIL FOR ANALYSIS *

Remove the surface accumulations of decaying leaves, etc., and take samples with a soil tube or auger to the desired depth. All samples of soils taken for analysis should be composite, and should be composed of representative samples taken from at least five different places in the field sample, each individual sample to be a column of uniform soil extending through the stratum sampled.

One composite sample should be taken from each important and distinctly different soil stratum to a depth of 40 in., or 1 m., including a composite sample from the arable stratum, or plowed soil, usually about 6 in., or 15 cm., deep.

If the plow line and subsoil coincide, and the subsoil is fairly uniform stratum to a depth of about 40 in., then only two composite samples need be taken — one of the arable soil and one of the subsoil. But if the subsoil line is lower than the plow line and not below 40 in., then both strata below the arable soil should be sampled, which

* *Bulletin No. 107* (Revised), Bureau of Chemistry, United States Department of Agriculture.

would make three composite samples from the field: one from the surface or arable soil, one from the subsurface soil (that is, from the stratum between the plow line and the true subsoil), and one from the true subsoil. Dry the samples in a well-aired, cool place.

APPARATUS USED IN THE ANALYSIS OF SOILS

1, weighing bottle; 2, measuring flasks (250 cc., 500 cc., and 1000 cc.); 3, water bath and tripod; 4, mortar and pestle; 5, suction flask, funnel, and Gooch crucible; 6, humus cylinder (500 cc.); 7, flask with reflux condenser; 8, sieve (holes 1 mm. in diameter)

PREPARATION OF SAMPLES FOR ANALYSIS

Prepare the laboratory sample by putting it through a sieve with round holes of 1 mm. diameter, using a rubber-tipped pestle to pulverize the lumps. Weigh, and discard the remaining material. Keep the sample in a cool place, and stopper to prevent change in condition. The fine soil

that passes through the holes of the sieve is used for the chemical analysis. Record the weight of the fine and the coarse soil as obtained.

Question

Why is the weight of the fine and the coarse soil desired?

Experiment No. 24

QUALITATIVE ANALYSIS OF SOIL

Place about 10 g. of soil in a beaker; then add 25 cc. of distilled water and 25 cc. of C. P. concentrated HCl. Cover the beaker with a watch glass and heat on a sand bath, wire gauze, or hot plate in the hood for two hours, replacing the acid if excessive evaporation takes place. Dilute with 50 cc. of distilled water, heat to boiling, and filter the solution, washing the residue with hot water. The insoluble residue consists mainly of silica and insoluble silicates. Evaporate the filtrate to dryness to remove silica from the solution, take up with 25 cc. hot water containing 5 cc. of HCl, and filter, washing the residue with hot water. Divide the filtrate into two portions: To Portion A add ammonium hydroxide (NH_4OH) until alkaline. The precipitate may contain $\text{Fe}(\text{OH})_3$ (red), $\text{Al}(\text{OH})_3$ (white and flocculent), and calcium phosphate ($\text{Ca}_3(\text{PO}_4)_2$) (white). Filter and test precipitate for iron and aluminium. Make the filtrate alkaline with NH_4OH and add the ammonium oxalate ($(\text{NH}_4)_2\text{C}_2\text{O}_4$) solution. A white precipitate of calcium oxalate (CaC_2O_4) may form, showing the presence of calcium. Make the filtrate from the calcium oxalate precipitation slightly acid with dilute HCl (1:1), concentrate to a volume of about 30 cc., add 5 cc. of sodium

ammonium hydrogen phosphate,* and then add gradually NH_4OH until the solution is distinctly alkaline. If the soil contains a very small quantity of magnesium, it may be necessary to let the solution stand for a short time to obtain a precipitate. Evaporate Portion B nearly to dryness; add 20 cc. of water, make alkaline with NH_4OH , and then slightly acid with dilute HNO_3 (1:1). Warm to approximately 70°C . and add a few cubic centimeters of ammonium molybdate $((\text{NH}_4)_2\text{MoO}_4)$. A yellow precipitate of ammonium phosphomolybdate $((\text{NH}_4)_3\text{PO}_4 \cdot 12 \text{MoO}_3)$ proves the presence of phosphates. Also test the original soil for carbonates. Make a detailed report of results, writing all equations illustrating the reactions.

Questions

1. In what chemical forms do the elements exist in a soil?
2. From what is the inorganic material or mineral matter of soils derived?
3. What elements are liable to be deficient in soils?

Experiment No. 25

DETERMINATION OF MOISTURE IN SOIL

Dry 2 to 5 g. of air-dried soil in a weighed watch glass for five hours at the temperature of boiling water, cool in a desiccator, and weigh rapidly to avoid absorption of moisture from the air. Heat again, and weigh at intervals of one hour until the material ceases to lose weight. Calculate and report the percentage of moisture in the soil.

* Prepared according to directions on page 89.

Experiment No. 26

DETERMINATION OF VOLATILE MATTER IN SOIL

Weigh out 2 to 5 g. of air-dried soil in a weighed porcelain crucible and heat to redness until all organic matter is burned. Cool the crucible containing the soil in a desiccator, and weigh. Continue the burning, cooling, and weighing until constant weight is obtained. If the soil contains appreciable quantities of carbonates, it should be moistened after cooling with a few drops of ammonium carbonate, dried, heated to dull redness to expel ammonium salts, cooled in desiccator, and again weighed. The loss in weight represents the hygroscopic moisture (see Exp. No. 25), water of combination, organic matter, ammonium salts, etc. From this loss, calculated as percentage, subtract the percentage of moisture as determined in Experiment No. 25 and obtain the percentage of matter volatile above 100°C . Make duplicate determinations. Calculate and report the percentage of volatile matter in the soil.

Question

Give the reason for the use of ammonium carbonate.

Experiment No. 27

DETERMINATION OF HUMUS IN SOIL

Place 10 g. of the soil sample in a Gooch crucible or on a filter paper in a funnel, and wash with dilute HCl (1 cc. concentrated acid to 40 cc. H_2O) until the filtrate after being made alkaline with NH_4OH gives no precipitate with ammonium oxalate $((\text{NH}_4)_2\text{C}_2\text{O}_4)$ solution.

Wash with distilled water until free from acid. Transfer the contents of the crucible or funnel into a glass-stoppered cylinder, with 500 cc. of a 4 per cent NH_4OH solution (1 cc. concentrated ammonia (sp. gr. .9) to 13 cc. of water), and allow it to remain, with occasional shaking, for twenty-four hours. During this time the cylinder should be inclined as much as possible without bringing its contents in contact with the stopper. Shake thoroughly and place the cylinder in a vertical position, add 2.5 g. of $(\text{NH}_4)_2\text{CO}_3$, and leave it for at least twelve hours to allow the sediment to settle. Filter the supernatant liquid (that is, remove the liquid above the sediment without stirring the sediment up; the filtrate will be colored, but should be free from any sediment), evaporate an aliquot portion (say 50 cc.) to dryness in a small porcelain evaporating dish on a water bath, dry at 100°C ., and weigh. Ignite the residue, cool the dish in a desiccator, and weigh again. Calculate and report the percentage of humus in sample.

Questions

1. What is humus ?
2. What are humates ?
3. What is humic acid ?
4. Give reasons for each step in the determination.
5. Of what value is the humus determination ?
6. Discuss the relation of organic matter in soils to fertility.

Experiment No. 28

DETERMINATION OF NITROGEN IN SOIL (NITRATES ABSENT)

Place 7 g. of soil in an 800 cc. Kjeldahl digestion flask with 30 cc. concentrated C. P. sulphuric acid, and continue as in Experiment No. 19. Make duplicate determinations. Calculate and report the percentage of nitrogen.

Experiment No. 29

TEST FOR ACIDITY OR ALKALINITY OF SOIL AND DETERMINATION OF LIME REQUIREMENTS

Place about 10 g. of soil in a beaker and add 75 cc. distilled water. After evaporating the filtrate to an approximate volume of 20 cc., stir for about five minutes and filter. Test the filtrate with phenolphthalein indicator to see if the solution is alkaline or acid. If the solution is alkaline, then determine whether it is due to Na_2CO_3 , Na_2SO_4 , or CaCO_3 in solution. If the solution is acid, then proceed as follows: Slake a small piece of lime (CaO) until it falls to a fine powder. This is best done by adding a few drops of water at a time, continuing until enough has been added to slake the lime thoroughly. With the slaked lime (Ca(OH)_2) make a saturated solution of limewater by the addition of approximately 250 cc. of warm water. Let the solution cool by standing; filter, and transfer the filtrate immediately to a glass-stoppered bottle. Titrate a portion of the clear solution with your standard HCl solution. Calculate and report the grams of CaO per cubic centimeter of limewater.

The standardized limewater may be used to determine the lime requirements or the acidity of the soil as follows: Weigh out two portions of 10 g. each, weighing only to the second decimal place; place them in two porcelain dishes of about 100 cc. capacity and moisten with about 75 cc. of distilled water; then to one add 5 cc. and to the other 10 cc. of the clear limewater solution from burette or pipette. Stir well, allow to stand about one hour, and then dry on a water bath. Add 50 to 75 cc. of distilled

water, stir thoroughly, and filter. About 30 cc. of the filtrate is then placed in a porcelain dish, three drops of neutral phenolphthalein added, and the solution boiled over a direct flame until about 10 cc. remains in the dish. The portion showing the alkaline color of phenolphthalein with the least amount of limewater has been saturated with lime. The portion showing no color is still acid or neutral.

Next weigh out four more portions of soil and treat with limewater in a similar way, using for the first 1 cc. more of limewater than was used in the portion that did not show alkaline, for the second 2 cc. more, etc. These are carried through as in the first series.

Note the least number of cubic centimeters of limewater that saturated the 10 g. of fine soil. From the data obtained, calculate and report the lime (CaO) required per acre of soil, using 2,000,000 lb. as the weight of an acre of soil 8 in. in depth. Also calculate the amount of ground limestone required per acre of soil.

Questions

1. Discuss soil acidity and alkalinity, including causes and correction. 2. If a soil solution tests acid with phenolphthalein as indicator when cold, and alkaline after boiling, what does this indicate? 3. Discuss the relation of the acidity of soils to soil fertility. 4. Give a complete discussion of alkali soils and the methods of correcting them.

Experiment No. 30

STRONG HYDROCHLORIC ACID (SP. GR. 1.115) DIGESTION OF SOIL. PREPARATION OF SOIL SOLUTION

Weigh out exactly 10 g. of soil, transfer to an Erlenmeyer flask of nonsoluble glass of 200 to 300 cc. capacity, and add 100 cc. of hydrochloric acid of constant boiling

point (sp. gr. 1.115) made, approximately, by diluting 58 cc. of ordinary concentrated HCl (sp. gr. 1.20) with 42 cc. of distilled water. The flask should be provided with a rubber stopper containing a glass tube about 20 in. in length. Place the flask containing the soil and acid in a water bath and digest for ten hours, shaking every hour while digesting. After the digestion is complete, allow the insoluble residue to settle, and decant the solution into an evaporating dish. Very small quantities of sediment passing over will do no harm. Wash the insoluble residue onto a filter with hot water, and continue the washing until free from chlorides, adding the washings to the original solution for evaporation. (It is advisable to let each portion in the filter paper run through each time before adding any more water.) After the addition of a few drops of nitric acid, to oxidize the organic matter in solution, evaporate the solution to dryness on a water bath. Take up with hot water and a few cubic centimeters of hydrochloric acid and again evaporate to complete dryness. When the evaporation is completed and the dish cooled, add a few drops of strong hydrochloric acid, sufficient only to saturate the residue. Add about 20 cc. of water, warm on a water bath to secure complete solution, and filter, washing until free of chlorides. Again evaporate the solution to dryness, to render insoluble any silica that may be in solution, take up with hydrochloric acid and water as before, and filter into a 500 cc. measuring flask, washing the residue free from chlorides. Make the solution in the measuring flask up to the mark with distilled water, shake thoroughly, and designate as Solution A.

Experiment No. 31

DETERMINATION OF IRON (Fe) IN SOIL SOLUTION BY VOLUMETRIC METHOD

Measure out, with a burette or pipette, two 50 cc. portions of Solution A, place in beakers, heat nearly to boiling, and add NH_4OH , drop by drop, until the solution is strongly alkaline. Boil for two minutes, and *filter while hot* through a properly prepared Gooch crucible, washing several times with hot water (save filtrate and washings for Exp. No. 32). Transfer the precipitate and asbestos to the same beaker from which it was filtered, and add 25 cc. of H_2SO_4 (1 : 4) to dissolve the precipitate. To this solution add a small amount of granulated zinc or zinc dust and boil until all traces of zinc have disappeared and the iron is completely reduced. Titrate the solution *immediately* with your standard KMnO_4 solution. If the reduced iron solution is allowed to stand exposed to the air before titrating, it will become oxidized. From the results obtained, calculate and report the percentage of iron in the soil sample soluble in the acid solution.

Questions

1. Give the reason why KMnO_4 acts as its own indicator.
2. What effect would a small amount of zinc in the solution have on the determination?
3. Write equations illustrating all the reactions, and explain each step.

Experiment No. 32

DETERMINATION OF CALCIUM (Ca) IN SOIL SOLUTION BY VOLUMETRIC METHOD

Evaporate the filtrate and washings from Experiment No. 31 to a volume of about 50 cc., make slightly alkaline with NH_4OH , and while boiling add ammonium oxalate $((\text{NH}_4)_2\text{C}_2\text{O}_4)$ solution *dropwise* until all the calcium is precipitated, then add 2 cc. in excess. Boil for five minutes, allow the precipitate to settle, and filter while hot on a filter paper, without using suction. Wash the precipitate and beaker free from soluble oxalates with *hot water*. Transfer filter paper and contents to the same beaker from which the precipitate was filtered, add 50 cc. of distilled water and 20 cc. of H_2SO_4 (1:1), heat *nearly* to boiling, and while still hot titrate with the standard KMnO_4 solution. Calculate and report the percentage of lime (CaO) in the soil sample soluble in the acid solution, and also its equivalent in calcium carbonate (CaCO_3).

NOTE. If preferred, the calcium can be determined directly upon Solution A in the following manner: Take 50 cc. of Solution A, make slightly alkaline with NH_4OH , and just clear with dilute HCl , *avoiding an excess*. Heat to boiling, add powdered ammonium oxalate or ammonium oxalate solution until the calcium is all precipitated, and let stand until cool (usually until the next period or overnight). Filter, wash, and titrate the precipitate as in the previous method.

Question

Write equations for all reactions taking place in the determination.

Experiment No. 33

DETERMINATION OF PHOSPHORIC ACID (P_2O_5) IN SOIL SOLUTION BY GRAVIMETRIC METHOD*

Measure out, with a pipette or burette, two 100 cc. portions of Solution A into beakers, and concentrate to a volume of about one fourth. Make alkaline with NH_4OH , then add 10 cc. in excess, and make *slightly* acid with HNO_3 (1:1), using a small piece of litmus paper in the solution as indicator. Avoid much *excess* of nitric acid. Gradually add to 20 cc. of the ammonium molybdate solution† the solution containing the phosphates, and place the beaker in the water bath at the temperature of 40° to $60^\circ C.$ (be sure to transfer the last trace of the phosphate solution from the beaker by rinsing with distilled water). When the precipitate has sufficiently settled, add a few cubic centimeters of the ammonium molybdate solution to the supernatant liquid, in order to be sure that all the phosphoric acid is precipitated. If any precipitate is produced, add more ammonium molybdate solution and repeat the operation until all the phosphoric acid is precipitated. After standing for three hours at a temperature not above $60^\circ C.$, filter on a small filter paper and wash with cold water until free from acid. Dissolve the ammonium phosphomolybdate precipitate on the filter paper by pouring a solution containing equal parts of ammonium hydroxide and hot water through the filter, receiving the solution in the beaker in which the first precipitation took place.

* If desired, phosphoric acid (P_2O_5) can be determined by optional method (Exp. No. 33 a).

† Prepared according to directions on page 89.

Be sure that all the yellow precipitate on the filter paper is dissolved, and do not allow the total volume of the filtrate to amount to more than 100 cc. Make the solution slightly acid with hydrochloric acid, using a small piece of litmus paper in the solution as indicator, and then *slightly* alkaline with NH_4OH . Cool the solution and add magnesia mixture * (from 10 to 15 cc.) from a burette or pipette, letting it run in at the rate of a drop per second, stirring the solution vigorously with a policeman, or rubber-tipped glass rod, at the same time. After fifteen minutes add 12 cc. of ammonium hydroxide (sp. gr. 0.90) and allow the solution to stand several hours — two hours is usually enough. (If more convenient, the solution may be covered with a watch glass and left until the next laboratory period before filtering.) Filter and wash the precipitate with 2.5 per cent ammonia (NH_3) solution until free from chlorides. After the precipitate is dried, place the filter paper and precipitate in a weighed crucible and ignite to whiteness. Cool in desiccator, weigh, and heat to constant weight. From the weight of magnesium pyrophosphate ($\text{Mg}_2\text{P}_2\text{O}_7$) calculate the weight and the percentage of phosphoric acid (P_2O_5) in the soil soluble in the acid solution.

Questions

1. Write equations for each step in the determination.
2. Discuss the term *phosphoric acid* (P_2O_5) as used.
3. Show the difference between a 2.5 per cent ammonia solution and a 2.5 per cent ammonium hydroxide solution.

* Prepared according to directions on page 91.

Experiment No. 33 a (Optional Method)

DETERMINATION OF PHOSPHORIC ACID (P_2O_5) IN SOIL SOLUTION BY VOLUMETRIC METHOD

Measure out, with a pipette or burette, two 100 cc. portions of Solution A into beakers, and concentrate to a volume of about one fourth. Make alkaline with NH_4OH , then add 10 cc. in excess, and make *slightly* acid with HNO_3 (1:1), using a small piece of litmus paper in the solution as indicator. Avoid much *excess* of HNO_3 . Gradually add to 20 cc. of the ammonium molybdate solution the solution containing the phosphates, and place the beaker in a water bath at the temperature of 40° to 60° C. (be sure to transfer the last trace of the phosphate solution from the beaker by rinsing with distilled water). When the precipitate has sufficiently settled, add a few cubic centimeters of the ammonium molybdate solution to the supernatant liquid, in order to be sure that all the phosphoric acid is precipitated. If any precipitate is produced, add more ammonium molybdate solution, and repeat the operation until all the phosphoric acid is precipitated. After the solution containing the precipitate stands for three hours at a temperature not above 60° C., filter on a small filter paper and wash with cold water until free from acid. Transfer the precipitate and filter paper containing the precipitate back into the same beaker in which it was precipitated. From the burette add the standard NaOH solution until the yellow precipitate is completely dissolved. Add about 25 cc. of distilled water and a few drops of phenolphthalein indicator.

1. Should the solution be colorless with the indicator added, continue the addition of the NaOH solution until a permanent pink color is obtained; that is, the end-point is reached.

2. Should the solution be colored after the indicator is added, that is, alkaline, titrate with your standard HCl solution until the color is discharged, and then titrate with your standard NaOH solution until the end-point is obtained.

In either case the total amount of NaOH used to *dissolve and titrate* the yellow precipitate and also the *total amount* of standard HCl used should be recorded. The precipitate is dissolved in the standard NaOH solution according to the following equation:

Two parts of ammonium phosphomolybdate

contain **1** part of phosphoric acid (P_2O_5).

Mol. wt. P_2O_5 : mol. wt. 46NaOH :: x : cc. of NaOH $\times$ the titre of the NaOH solution

x = grams of phosphoric acid (P_2O_5) in the aliquot portion of the soil solution. From the data obtained, the titre of the NaOH solution, and the equation, calculate and report the percentage of phosphoric acid (P_2O_5) in the soil soluble in the acid solution.

Question

Give the reason for each step in the determination and write equations illustrating the reactions.

Experiment No. 34

DETERMINATION OF POTASH (K_2O) IN SOIL SOLUTION BY THE USE OF PLATINUM SOLUTION*

Measure out, with a burette or pipette, two 100 cc. portions of Solution A, place in beakers, heat nearly to boiling, and add NH_4OH , *drop by drop*, until the solution is strongly alkaline. Cover the beaker and boil the solution for about one minute; if no ammonia is given off (detect by smelling), more is added, *drop by drop*, until it can be detected. Do not allow the precipitate to settle, but stir and filter immediately while hot, washing it thoroughly with *hot water*. Evaporate the filtrate and washings to complete dryness, heat below redness to expel ammonium salts, dissolve in about 25 cc. of hot water, add about 5 cc. of saturated barium hydroxide, and heat to boiling. Let the solution stand until the precipitate settles, and test the supernatant liquid with a drop of barium hydroxide solution to be sure that precipitation is complete. When the precipitation is complete, filter and wash the residue thoroughly with hot water. While the solution is boiling add ammonium hydroxide and ammonium carbonate to precipitate the barium. Allow the solution to stand for a short time on a water bath, filter, wash the precipitate thoroughly with hot water, and evaporate filtrate and washings to dryness. Expel the ammonium salts by heating at a low red heat as before, dissolve the residue in about 20 cc. of water, add about 2 cc. of ammonium

* If desired, potash can be determined by optional methods (Exps. Nos. 34 a, 34 b).

hydroxide and a drop of ammonium carbonate solution, let stand on a water bath for a few minutes, and filter into an evaporating dish. Evaporate filtrate and washings nearly to dryness, add 1 cc. of dilute H_2SO_4 (1:1), evaporate to dryness, and ignite to whiteness. As all the potassium is in the form of sulphate, no loss need be apprehended by volatilization of potash, and a full red heat must be maintained until the residue is perfectly white. Dissolve the residue in about 15 cc. of water, add a few drops of hydrochloric acid, and platinum solution (H_2PtCl_6) * in slight excess (avoid the use of too large an excess, as the reagent is very expensive). Evaporate on a water bath to a thick paste, and treat the residue with 80 per cent alcohol (sp. gr. 0.8645). If enough platinum solution has been added to precipitate all the potassium, the alcoholic solution will be slightly colored. If the alcohol is not slightly colored, it must be evaporated off, the precipitate redissolved in water containing a few drops of HCl , more platinum solution added, and again evaporated to a thick paste and treated with alcohol as before. If the alcoholic solution is not colored, owing to an excess of platinum solution, do not add more platinum solution directly to the alcoholic solution, but evaporate off the alcohol first. *Also avoid the absorption of ammonia by the solution.* After the precipitate is treated with the 80 per cent alcohol, filter into a properly prepared and *weighed* Gooch crucible, wash thoroughly with 80 per cent alcohol, then with 10 cc. of the special ammonium chloride solution † to remove impurities. Repeat washing with the special ammonium chloride solution about five times, and

* Prepared according to directions on page 90.

† Prepared according to directions on page 90.

again wash thoroughly with 80 per cent alcohol. Dry the precipitate for thirty minutes at the temperature of boiling water, cool in desiccator, and weigh. The precipitate is potassium platinic chloride (K_2PtCl_6), and should be perfectly soluble in water (which gives a means of checking the results if desired). For the conversion of potassium platinic chloride to potassium oxide (K_2O) use the factor 0.1941. Calculate and report the percentage of potash (K_2O) in the soil soluble in the acid solution. (Save all filtrates and potassium platinic chloride precipitates so as to recover the platinum.)

Questions

1. How is the factor 0.1941 obtained? 2. Write equations illustrating each step in the determination.

Experiment No. 34 *a* (Optional Method)

DETERMINATION OF POTASH (K_2O) IN SOIL SOLUTION BY VOLUMETRIC METHOD *

Measure out, with a burette or pipette, two 100 cc. portions of Solution A, place in beakers, heat to boiling, add NH_4OH , *drop by drop*, until the solution is strongly alkaline, and a few cubic centimeters of ammonium oxalate solution. Cover the beaker and boil the solution for about one minute; if no ammonia is given off (detect by smelling), more is added, drop by drop, until it can be detected. Do not allow the precipitate to settle, but stir and filter immediately while hot, washing it thoroughly with hot water.

* Most of the points in the manipulation were taken directly from a method suggested by O. M. Shedd for the determination of total potassium in soils, *Journal of Industrial and Engineering Chemistry*, Vol. I, No. 5, May, 1909.

Evaporate the filtrate and washings nearly to dryness in an evaporating dish, add 1 cc. of dilute H_2SO_4 (1:1), evaporate to dryness, and ignite to whiteness by maintaining a full red heat until the residue is perfectly white.

Dissolve the residue in hot water, filter if necessary to remove any insoluble material, acidify the clear solution slightly with acetic acid, and evaporate to a volume of about 10 or 15 cc.* Ten cubic centimeters or a liberal excess of the cobaltinitrite reagent, prepared according to Adie and Wood, are added slowly, so that the precipitate may not be too finely divided, and the liquid evaporated off on the water bath to a sirupy consistency, becoming solid on cooling. It is important not to heat longer than is necessary. After cooling, the soluble matters are dissolved in about 25 cc. of cold water (which should give a brown solution, showing excess of reagent), the solution decanted through a carefully prepared Gooch crucible,† and this operation repeated until the dish and any precipitate remaining in it have been thoroughly washed.‡ Be sure that all the precipitate is completely removed from the dish. After washing the filter, the contents of the crucible (the asbestos with the precipitate) are transferred to a 500 cc. beaker and well broken up by stirring with a glass rod in a little water. If any of the precipitate adheres to the Gooch crucible so that it cannot be washed off, the crucible also is to be put into the dish. A measured excess

* When the reagent is added to a dilute solution, it is decomposed before the potassium salt is precipitated. In small volumes this does not happen.

† The asbestos pulp for making the filter should be just fine enough to hold the precipitate and be free from very fine particles.

‡ A half-saturated solution of common salt may be used instead of water if there is trouble in filtering the precipitate.

of standard potassium permanganate solution* (usually 20–40 cc.) is now run in, and the whole diluted to about eight or ten times the volume of permanganate added, the dish covered, and its contents heated to boiling over a free flame or on a hot plate, with frequent stirring for about ten minutes, or until the potassium cobaltinitrite is oxidized completely. It has been found that the oxidation requires a somewhat longer time than the five to eight minutes recommended by Drushel, apparently because it is hard to separate the yellow potassium precipitate from the asbestos so that the permanganate can come in contact with it. When the oxidation is complete — as indicated by the darkening of the solution and the separation of the manganese hydroxide — about 15 cc. of dilute sulphuric acid (1:7) are added and allowed to act three or four minutes to favor oxidation of the last traces of cobaltinitrite.† A measured excess of the standard oxalic acid‡ containing 50 cc. of concentrated sulphuric acid to the liter is then run in, and the liquid kept at the same temperature until all the manganic hydroxide has been dissolved and the solution is colorless. At this point it will be seen by the absence or presence of the yellow potassium compound whether the oxidation of the cobaltinitrite precipitate was complete. The excess of oxalic acid is now titrated with the standard permanganate solution.§ The total volume of permanganate solution used, less that equivalent to the oxalic acid added, gives the amount used up in oxidizing

* See Experiment No. 15 (approximate N/10 solution).

† The sulphuric acid is not to be added at first, along with the permanganate, as the action would be very rapid and some cobaltinitrite might escape oxidation.

‡ Prepared according to directions on page 90.

§ See Experiment No. 15.

the cobaltinitrite; and this, multiplied by the appropriate factor, gives the weight of potassium obtained. One cubic centimeter of N/10 permanganate solution is equivalent to 0.000711 g. of K, or 0.000856 g. of K_2O . If the potassium permanganate solution used for the titration is not exactly N/10, then it is necessary to calculate the appropriate factor to be used. It is also necessary to carry out a blank experiment, under the same conditions as the analysis, using the same quantities of the reagents, and to subtract the small amount of permanganate solution consumed from that found in the analysis. From the results obtained, calculate and report the percentage of potash (K_2O) in the sample of soil soluble in the acid solution.

Question

How is the factor for the K equivalent of 1 cc. N/10 KMO_4 obtained?

Experiment No. 34 b (Optional Method)

DETERMINATION OF POTASH (K_2O) IN SOIL SOLUTION BY THE USE OF PLATINUM SOLUTION*

Measure out, with a burette or pipette, two 100 cc. portions of Solution A into glazed porcelain evaporating dishes, add 10 cc. concentrated C. P. hydrochloric acid, and evaporate to dryness. Take up with 10 to 15 cc. of distilled water, add about 5 cc. of platinum chloride solution and 2 or 3 cc. of hydrochloric acid, and evaporate to dryness or to a thick paste on a water bath. Remove the dish from the water bath and let stand until it is

* This method is a modification of the method proposed by C. C. Moore, *Journal of American Chemical Society*, Vol. XX (1898), p. 340, and of the method used in the laboratories of the Texas Experiment Station.

perfectly cold. Add from 10 to 30 cc. of "acid alcohol,"* according to the amount of precipitate in the dish. All other materials beside the precipitate of potassium platinic chloride should be completely dissolved. If there is heat evolved when the acid alcohol is added, more alcohol is added to cool the solution, as this reaction often forms a white insoluble substance which would ruin the results. If enough platinum solution has been added to precipitate all the potassium, the alcoholic solution will be slightly colored. If the alcohol is not slightly colored, it has to be evaporated off, the precipitate redissolved in distilled water containing a few drops of hydrochloric acid, more platinum solution added, and again evaporated to a thick paste and the solution treated with acid alcohol, as before. If the alcoholic solution is not colored, owing to an excess of platinum solution, do not add more platinum solution directly to the alcoholic solution, but evaporate off the alcohol first. After the precipitate is treated with acid alcohol, filter by decantation into a properly prepared and *weighed* Gooch crucible; wash once with acid alcohol, then with 95 per cent alcohol until the alcohol wash does not dissolve any more colored material of any kind; pour the washings through the crucible each time, but leave the precipitate in the evaporating dish as far as possible. Add 10 cc. of special ammonium chloride solution† to the precipitate in the dish, let stand a few minutes so as to dissolve impurities, and pour off the solution through the Gooch crucible. Repeat the washing with the special ammonium chloride solution about five times and again

* 10 cc. of concentrated C. P. hydrochloric acid solution added to 100 cc. of 95 per cent alcohol.

† Prepared according to directions on page 90.

wash thoroughly with 95 per cent alcohol. Transfer the potassium platinic chloride precipitate from the dish to the Gooch crucible by the use of 95 per cent alcohol, washing the side of the crucible carefully so as to remove any adhering solution of ammonium chloride. Dry the precipitate for thirty minutes at the temperature of boiling water, cool in desiccator, and weigh. The precipitate is potassium platinic chloride (K_2PtCl_6), and should be perfectly soluble in water (which gives a means of checking the results if desired). For the conversion of platinic chloride to potassium oxide (K_2O) use the factor 0.1941. Calculate and report the percentage of potash (K_2O) in the soil soluble in the acid solution. (Save all filtrates and potassium platinic chloride precipitates so as to recover the platinum.)

NOTE. "Available" phosphoric acid and potash in soils can be determined by digestion with 1 per cent citric acid (Dyer's method). *Journal of American Chemical Society*, Vol. LXVI (1894), p. 115; Lunge, *Technical Methods of Chemical Analysis*, Vol. I, p. 852.

PART IV

ANALYSIS OF FERTILIZERS

PREPARATION OF SAMPLE OF FERTILIZER FOR ANALYSIS *

Grind the sample fine enough to pass through a sieve having circular perforations 1 mm. in diameter, and then mix thoroughly. Perform the grinding and sifting as rapidly as possible to avoid loss or gain during the operation. After the sample is prepared and thoroughly mixed, it should be kept in a well-stoppered bottle. Enough sample should be prepared for all the determinations.

Experiment No. 35

DETERMINATION OF MOISTURE IN FERTILIZER

Weigh out exactly 2 g. of the fertilizer in a weighed watch glass and dry for three hours at the temperature of boiling water; cool in desiccator, and weigh rapidly. Heat again at intervals of one-half hour until the material ceases to lose weight. Make duplicate determinations. Calculate and report the percentage of moisture in the sample of fertilizer.

* *Bulletin No. 107* (Revised), Bureau of Chemistry, United States Department of Agriculture.

Experiment No. 36

PREPARATION OF A STANDARD NaOH SOLUTION FOR PHOSPHORIC ACID (P_2O_5) DETERMINATION

Make up a solution (500 cc.) of sodium hydroxide (NaOH) so that 10 cc. of this solution should neutralize 16.2 cc. of an *exactly* $N/5$ HCl solution. If your standard hydrochloric acid solution is not *exactly* $N/5$, then calculate the number of cubic centimeters of *your HCl solution*

COMBINATION ELECTRIC WATER BATH AND HOT PLATE

which will be *equivalent* to 16.2 cc. of an *exactly* $N/5$ HCl solution. Standardize the NaOH solution so that 10 cc. of it will exactly neutralize the calculated number of cubic centimeters of your standard acid. One cubic centimeter of the NaOH solution will then be equivalent to .001 g. of phosphoric acid (P_2O_5).

Question

Show that 1 cc. of the NaOH solution is equivalent to .001 g. of P_2O_5 .

NOTE. For equation see Experiment No. 33 a.

Experiment No. 37

DETERMINATION OF TOTAL PHOSPHORIC ACID (P_2O_5) IN FERTILIZER

Weigh out 2 g. of the sample of fertilizer and place it in a beaker with 15 to 20 cc. of concentrated hydrochloric acid and from 3 to 10 cc. of concentrated nitric acid, and digest from 20 to 30 minutes *under the hood*. After the solution is complete, cool, add 25 cc. of distilled water, filter, and wash the residue thoroughly, allowing the washings and the original solution to run into a 250 cc. *measuring flask*. Make up to the mark with distilled water, stopper the flask, and shake thoroughly. Measure out, with a burette or pipette, two 25 cc. portions into beakers for analysis, and to each portion add 25 cc. of distilled water. Make alkaline with NH_4OH , adding 10 cc. in excess, and then *slightly* acid with HNO_3 (1:1), using a small piece of litmus paper in the solution as indicator. Avoid much excess of HNO_3 . Warm the solution to the temperature of 60° to 65° C. by standing the beakers in a pan containing warm water. After the solution has reached the required temperature add 30 cc. of ammonium molybdate solution; stir, and let stand for fifteen minutes in the warm water at 60° to 65° C. Filter *at once* and wash twice with water by *decantation*, by pouring the solution through the filter, using 25 to 30 cc. of water each time, agitating the precipitate thoroughly, and allowing it

to settle. Transfer to the filter paper and wash with cold water until washings are free from acid. (Test by means of litmus paper.) Transfer the filter paper containing the precipitate back into the beaker in which it was precipitated. From the burette add your standard NaOH solution (prepared for the P_2O_5 determination) until the yellow precipitate is *completely* dissolved. Add about 25 cc. of distilled water and a few drops of phenolphthalein indicator.

1. Should the solution be colorless with the indicator added, continue the addition of NaOH solution until a permanent pink color is obtained; that is, the end-point is reached.

2. Should the solution be colored after the indicator is added, that is, alkaline, titrate with your standard HCl solution until the color is discharged, and then titrate with your standard NaOH solution until the end-point is obtained.

In either case the total amount of NaOH used to *dissolve and titrate* the yellow precipitate and also the *total amount* of standard HCl used should be recorded. From the data obtained calculate and report the percentage of phosphoric acid (P_2O_5) in the sample of fertilizer.

Questions

1. Give the reason for each step in the determination and write equations for the reactions. 2. Define raw rock phosphate, basic slag, acid phosphate, water-soluble phosphoric acid, reverted phosphoric acid, insoluble phosphoric acid, and give their chemical compositions and methods of preparation.

Experiment No. 38

DETERMINATION OF WATER-SOLUBLE PHOSPHORIC ACID (P_2O_5) IN FERTILIZER

Weigh out 2 g. of the sample, place it on a 9 cm. filter paper, and leach with successive small portions of distilled water into a 250 cc. *measuring flask* (allowing each portion of water to run through before adding more) until the filtrate measures nearly 250 cc. If the filtrate is turbid, add a few drops of concentrated HNO_3 . Make up to the mark with distilled water and shake the solution thoroughly. Measure out, with burette or pipette, two 25 cc. portions into beakers and proceed as in Experiment No. 37. *Save the residue in the filter paper for Experiment No. 39.*

Experiment No. 39

DETERMINATION OF CITRATE-SOLUBLE PHOSPHORIC ACID (P_2O_5) IN FERTILIZER

Heat 100 cc. of *strictly neutral* ammonium citrate solution* (sp. gr. 1.09) to $65^\circ C.$ in a 200 to 300 cc. Erlenmeyer flask placed in a warm water bath, keeping the flask loosely stoppered. When the citrate solution has reached the temperature of $65^\circ C.$, drop into it the filter paper containing the *leached residue* from the water-soluble phosphoric acid determination (Exp. No. 38), close tightly with a smooth rubber stopper, and shake violently until the filter paper is reduced to a pulp. Place the flask in the bath, and maintain it at such a temperature (about $67^\circ C.$)

* Prepared according to directions on page 91.

that the contents of the flask will stand exactly at 65° C. Shake the flask every five minutes. At the expiration of *exactly thirty minutes* from the time the filter with contents was introduced, remove the flask from the bath and filter as rapidly as possible. Wash the *residue and flask thoroughly* with distilled water heated to the temperature of 65° C. Return the filter with contents to the Erlenmeyer flask, add 30 cc. of concentrated HNO_3 and 10 cc. of concentrated HCl , and digest under the hood until phosphates are dissolved; that is, about twenty or thirty minutes. Dilute with 50 cc. of distilled water, and filter into a 250 cc. *measuring flask*, washing the residue thoroughly. Make up with distilled water to the mark, shake thoroughly, and measure out, with burette or pipette, two 25 cc. portions into two beakers. Then proceed as in Experiment No. 37. The total phosphoric acid minus the sum of the water-soluble and citrate-insoluble gives the citrate-soluble phosphoric acid. Calculate and report the percentage of citrate-insoluble and citrate-soluble phosphoric acid (P_2O_5) in the sample of fertilizer.

Questions

1. Give the formulas for molybdic acid, ammonium molybdate, ammonium phosphomolybdate, and citrate-soluble phosphoric acid.
2. Write the equation for the reaction of ammonium phosphomolybdate and NaOH .
3. Discuss the solubility of monocalcium, dicalcium, and tricalcium phosphate.
4. Define "available" phosphoric acid.

Experiment No. 40

DETERMINATION OF POTASH (K_2O) IN A MIXED FERTILIZER BY THE USE OF PLATINUM SOLUTION *

Boil 5 g. of the fertilizer sample with 300 cc. of distilled water thirty minutes. Add to the hot solution a slight excess of NH_4OH and then sufficient ammonium oxalate solution to precipitate all the lime present. Cool, dilute to 500 cc. with distilled water in a *measuring flask*, shake thoroughly, and filter through a *dry* filter. Evaporate in an evaporating dish 100 cc. of the solution (measured exactly and corresponding to 1 g. of the sample) nearly to dryness, add 1 cc. of dilute sulphuric acid (1:1), evaporate to dryness (being careful not to lose anything by spattering), and ignite to whiteness. As all the potassium is in the form of sulphate, no loss need be apprehended by volatilization of potash, and a full red heat must be maintained until the residue is perfectly white. Dissolve the residue in hot water and filter if any insoluble material remains. Add to the clear solution a few drops of HCl , and platinum solution (H_2PtCl_6)[†] in slight excess. Evaporate on a water bath to a thick paste and treat the residue with 80 per cent alcohol (sp. gr. 0.8645). If enough platinum solution has been added to precipitate all the potassium, the alcoholic solution will be slightly colored. If the alcohol added to the residue is not slightly colored, the alcohol has to be evaporated off, residue redissolved in water containing a few drops of HCl , more platinum

* Potash can be determined by optional method (Exp. No. 40 a).

† Prepared according to directions on page 90.

solution added, and the solution again evaporated to a thick paste and treated with alcohol. Do not add more platinum solution to the alcoholic solution before evaporating off the alcohol. *Avoid the absorption of ammonia by the solution.* After the residue is treated with 80 per cent alcohol, filter into a properly prepared and *weighed* Gooch crucible and wash the precipitate thoroughly with 80 per cent alcohol, then with 10 cc. of the special ammonium chloride solution,* to remove impurities. Repeat washing with 10 cc. portions of special ammonium chloride solution about five times, and again wash thoroughly with the alcoholic solution. Dry the precipitate for thirty minutes at the temperature of boiling water, cool in desiccator, and weigh. The precipitate is potassium platonic chloride (K_2PtCl_6) and should be perfectly soluble in water (which gives a means of checking the results if desired). For the conversion of potassium platonic chloride (K_2PtCl_6) to potassium oxide (K_2O) use the factor 0.1941. From data obtained, calculate and report the percentage of potash (K_2O) in the sample of fertilizer. (Save all the filtrates and potassium platonic chloride precipitates so as to recover the platinum.)

Experiment No. 40 a (Optional Method)

DETERMINATION OF POTASH (K_2O) IN MIXED FERTILIZER BY VOLUMETRIC METHOD

Measure out into evaporating dishes, with a burette or pipette, two 25 cc. portions of the filtered potassium solution prepared in Experiment No. 40. Evaporate each portion nearly to dryness in an evaporating dish, add 1 cc.

* Prepared according to directions on page 90.

of dilute sulphuric acid (1:1), evaporate to dryness, and ignite to whiteness by maintaining a full red heat until the residue is perfectly white. Dissolve the residue in hot water, filter if any remains undissolved, acidify the clear solution slightly with acetic acid, and proceed in the same manner as in Experiment No. 34 *a*. From data obtained calculate and report the percentage of potash (K_2O) in the sample of fertilizer.

Experiment No. 41

DETERMINATION OF NITROGEN IN FERTILIZERS (NITRATES PRESENT)

Weigh out *exactly* 1 to 2 g. of the sample and transfer to an 800 cc. Kjeldahl flask. Add 30 cc. salicylic acid solution* and 5 g. of sodium thiosulphate. Heat over a low flame until all danger of frothing has passed, cool, and then add about 10 g. of K_2SO_4 and about .5 g. copper sulphate ($CuSO_4 \cdot 5 H_2O$). Continue the digestion until the material is completely oxidized. Make the distillation and titration as given in Experiment No. 19. Make duplicate determinations. From data obtained, calculate and report the percentage of nitrogen in the sample of fertilizer.

Questions

1. Explain the use of salicylic acid and sodium thiosulphate. Write equations illustrating. 2. Explain the other steps in the determination.

* Prepared according to directions on page 91.

PART V

ANALYSIS OF INSECTICIDE AND FUNGICIDE

Experiment No. 42

PREPARATION AND STANDARDIZATION OF SOLUTION FOR DETERMINATION OF ARSENIOS OXIDE (As_2O_3) IN PARIS GREEN

Starch solution for indicator. Place 1 g. of starch in 10 cc. of cold distilled water to separate the granules, and then pour this mixture into 100 cc. of boiling water. Boil for five minutes, stirring continuously.

Standard iodine solution. Dissolve about 3.5 g. of iodine in 200 cc. of distilled water in which has been dissolved from 8 to 10 g. of pure potassium iodide (KI). Dilute to a volume of 500 cc. with distilled water and shake thoroughly.

Before standardizing be sure that all the iodine is in solution. Dissolve *exactly* 1 g. of pure arsenious oxide (As_2O_3) in 50 cc. of HCl (1:1), heating *rapidly* if necessary to bring all the arsenic into solution. (*Do not boil the solution.*) Cool and make up to a volume of 250 cc. in a *measuring flask*. Perform the titration as follows: Measure out, with a burette or pipette, two portions of 25 cc. of the arsenious oxide (As_2O_3) solution into porcelain evaporating dishes (about 6 in. in diameter) or into

large beakers, add about 300 cc. of distilled water, and sodium bicarbonate (NaHCO_3) in *slight* excess. Add the iodine solution from a burette, using the starch solution as an indicator. The titration is complete when you obtain the first permanent blue color. Make at least two titrations. Calculate the strength of the iodine solution in terms of arsenious oxide (As_2O_3).

Question

Why should the hydrochloric acid solution of arsenious oxide not be boiled?

Experiment No. 43

DETERMINATION OF TOTAL ARSENIOS OXIDE (As_2O_3) IN PARIS GREEN

To determine the total arsenious oxide in Paris green use 2 g. and proceed *exactly* as with the arsenious oxide in the standardization of iodine solution (Exp. No. 42). Make duplicate determinations. Calculate and report the percentage of arsenious oxide (As_2O_3) in the sample of Paris green.

Questions

1. Is Paris green a compound or a mixture? 2. If a compound, what is its formula? 3. To what is the As_2O_3 oxidized by the iodine? 4. Give equations for the reaction. 5. How much As_2O_3 can Paris green contain and still be safe as an insecticide? 6. Give a simple test for the purity of Paris green.

Experiment No. 44

DETERMINATION OF WATER-SOLUBLE ARSENIOS OXIDE (As_2O_3) IN PARIS GREEN

Place 1 g. of Paris green (weighed exactly) in a large flask with *exactly* 500 cc. of distilled water (previously boiled to expel carbon dioxide and then cooled to room temperature). Stopper the flask, shake thoroughly, and let stand for one week, shaking as often as convenient. At the end of this time filter the solution through a *dry filter*. Dilute 100 cc. of the filtrate with 100 cc. of distilled water, add sodium bicarbonate (NaHCO_3) in *slight* excess, and titrate with your standard iodine solution in the same manner as in Experiment No. 42, using the starch solution as indicator. Make duplicate titrations. Calculate and report the percentage of water-soluble arsenious oxide (As_2O_3) in the sample of Paris green.

ANALYSIS OF LEAD ARSENATE*

Preparation of sample. If the sample is in the form of a paste (as it usually is), dry the whole sample to constant weight at the temperature of boiling water and record the results as total moisture. Grind the dry sample (which will gain a small amount of moisture during grinding) to a fine powder and determine the various constituents as follows:

* These methods are modifications of methods proposed by Haywood, *Bulletin No. 105*, Bureau of Chemistry, United States Department of Agriculture (1907), p. 165.

Experiment No. 45

DETERMINATION OF MOISTURE IN LEAD ARSENATE

Heat 2 g. of the sample in the water oven at the temperature of boiling water for eight hours or in the hot-air oven at 110°C . for from five to six hours or till constant weight is obtained. Make duplicate determinations. Calculate and report the percentage of moisture in the sample of lead arsenate.

Experiment No. 46

DETERMINATION OF TOTAL LEAD OXIDE IN LEAD ARSENATE

Dissolve 2 g. of the sample in about 80 cc. of water and 15 cc. of concentrated nitric acid on the steam or water bath; transfer the solution to a 250 cc. measuring flask and make up to the mark. To 50 cc. of the solution add 3 cc. of concentrated sulphuric acid; evaporate on the steam bath to a sirupy consistency and then on a hot plate till white fumes appear and all nitric acid has been driven off. Add 50 cc. of water and 100 cc. of 95 per cent alcohol, let stand for several hours, and filter off the supernatant liquid; wash about ten times with acidified alcohol (water 100 parts, 95 per cent alcohol 200 parts, and concentrated sulphuric acid 3 parts) and then with 95 per cent alcohol* till free from sulphuric acid. Dry, transfer as much as possible of the precipitate from the paper into a weighed crucible, and ignite at a low red

* Prepared according to directions on page 92.

heat. Burn the paper in a separate porcelain crucible, and treat the residue first with a little nitric acid, which is afterwards evaporated off, and then with a drop or two of sulphuric acid. Ignite, weigh, and add this weight to the weight of the precipitate previously removed from the paper for the amount of the lead sulphate. Calculate and report the percentage of lead oxide in the sample of lead arsenate.

Question

Write equations for the chemistry of each step in the determination.

Experiment No. 47

DETERMINATION OF WATER-SOLUBLE LEAD OXIDE IN LEAD ARSENATE

Weigh out 2 g. of the lead arsenate, place in a flask with 2000 cc. of carbon-dioxide-free water, and let stand for a week, shaking as often as convenient (eight times a day if possible). Filter through a *dry filter* and use aliquots (200 to 400 cc.) of this solution for determining soluble lead oxide and arsenic oxide (As_2O_5); determine lead as described in Experiment No. 46 for total lead oxide, using the same relative proportions of sulphuric acid, water, and alcohol, but keeping the volume as small as possible. Make duplicate determinations. Calculate and report the percentage of water-soluble lead oxide in the sample of lead arsenate.

Experiment No. 48

DETERMINATION OF TOTAL ARSENIC OXIDE (As_2O_5) IN LEAD ARSENATE

Transfer 100 cc. of the nitric acid solution of the sample, prepared as in the determination of lead (Exp. No. 46), to a porcelain dish, add 6 cc. of concentrated sulphuric acid, evaporate on the water bath to a sirupy consistency and then on a hot plate until the appearance of white fumes of sulphuric acid. Wash into a 100 cc. flask with water, make up to the mark with distilled water, filter through a *dry filter*, and use 50 cc. aliquot parts for further work. Transfer this to an Erlenmeyer flask of 400 cc. capacity, add 4 cc. of concentrated sulphuric acid and 1 g. of potassium iodide, dilute to about 100 cc., and boil until the volume is reduced to about 40 cc. Cool the solution under running water, dilute to about 300 cc., and *exactly* use up the iodine set free and still remaining in the solution with a few drops of approximately tenth-normal sodium thiosulphate solution. *Be careful that an excess of sodium thiosulphate is not used.* Wash the mixture into a large beaker, make alkaline with sodium carbonate, and slightly acidify with dilute sulphuric acid, using up all the sodium carbonate; then make alkaline with an excess of sodium bicarbonate. Titrate the solution with your standard iodine solution until a blue color appears, using the starch solution as indicator. Calculate and report the percentage of arsenic oxide (As_2O_5) in the sample of lead arsenate.

Questions

1. Discuss the commercial preparation of lead arsenate and varieties.
2. Name the desirable properties of an insecticide.
3. Write equations for the chemistry of each step in the determination.

Experiment No. 49

DETERMINATION OF WATER-SOLUBLE ARSENIC OXIDE (As_2O_5) IN LEAD ARSENATE

For this determination use 200 to 400 cc. of the water extract obtained under the determination of soluble lead oxide (Exp. No. 47). Add 0.5 cc. of sulphuric acid and evaporate to a sirupy consistency, then heat on a hot plate until white fumes appear. Add a very small amount of distilled water, and filter through a small filter paper to remove the lead, using as little wash water as possible. Place this filtrate in an Erlenmeyer flask, and determine arsenic, as described above, for total arsenic oxide, using the same amount of reagents and the same dilutions. Calculate and report the percentage of water-soluble arsenic oxide (As_2O_5) in the sample of lead arsenate.

Experiment No. 50

TESTING BORDEAUX MIXTURE FOR SOLUBLE COPPER

Take a small piece of quicklime (CaO) and slake it with water. Weigh out on the rough balance about 5 g. of copper sulphate ($\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$) and dissolve in about 100 cc. of water. Add the milk of lime to the copper sulphate solution until you cannot obtain a test with potassium ferrocyanide ($\text{K}_4\text{Fe}(\text{CN})_6$).^{*} (The test for

^{*} Prepared according to directions on page 92.

soluble copper should be made by using small filtered portions taken from the original mixture.) This is known as Bordeaux mixture. Take a portion of the mixture, dilute it with water, and pass carbon dioxide into it for about fifteen minutes. Again test the solution for soluble copper.

Questions

1. Explain your results. 2. Also make the test for soluble copper by adding a drop of dilute ammonia to the clear solution, noting the blue copper hydroxide. Add more ammonia and observe the soluble blue compound. 3. How does the carbon dioxide of the air cause bad effects with Bordeaux mixture? 4. How could you prevent it? 5. Discuss the composition of Bordeaux mixture and the killing of foliage by its use as a spray. 6. With what insecticides can it be used?

PART VI

ANALYSIS OF MILK

Experiment No. 51

DETERMINATION OF SPECIFIC GRAVITY OF MILK

The sample of milk is thoroughly mixed and poured into a cylinder or hydrometer jar. Determine first the temperature of the milk and then, by means of a hydrometer or lactometer, determine the specific gravity. The hydrometer or lactometer should be gently lowered into the milk and the reading observed from the top of the meniscus. The temperature of the milk should be adjusted to 15.5°C . before the reading is made, or the correction for the temperature should be made so as to record the specific gravity reading at the temperature of 15.5°C . or 60°F . The Quevenne and New York Board of Health lactometers are used to the largest extent for this determination. To prevent the milk from souring before the other determinations are made, add 1 cc. of formalin (40 per cent solution of formaldehyde) to one pint of the milk, and keep the bottle stoppered.

Questions

1. What is specific gravity?
2. Name four ways of determining specific gravity of a liquid.
3. Explain the effect of

watering and skimming (and the two combined) upon the specific gravity.

References

LEACH. Food Inspection and Analysis.

RICHMOND. Dairy Chemistry.

VAN SLYKE. Modern Methods of Testing Milk and Milk Products.

WILEY. Agricultural Analysis, Vol. III.

Experiment No. 52

DETERMINATION OF TOTAL SOLIDS IN MILK

Measure out, with a burette or pipette, 10 cc. of milk of known specific gravity into a *tared* flat-bottomed dish of not less than 5 cm. diameter. Evaporate to dryness on a water bath, and dry in water oven for three hours at the temperature of boiling water. Cool in desiccator and weigh rapidly. Heat again at intervals of one-half hour until the material ceases to lose weight. Make duplicate determinations. Calculate and report the percentage of total solids in the sample of milk.

Questions

1. What does the total solids of milk contain? 2. The milk residue should be nearly pure white (a brownish color shows decomposition). Explain. 2. What effect would the souring of milk have on the content of the total solids in the milk? Explain.

Experiment No. 53

DETERMINATION OF ASH IN MILK

Measure out, with a burette or pipette, two 25 cc. portions of milk into *weighed* porcelain dishes. Add 5 cc. of concentrated HNO_3 , evaporate to dryness, and ignite at

a temperature just below redness until the ash is free of carbon. Cool in desiccator and weigh. Make duplicate determinations. Calculate and report the percentage of ash in the sample.

Question

What does the ash of milk contain?

Experiment No. 54

DETERMINATION OF FAT IN MILK BY THE WERNER-SCHMIDT METHOD

Transfer 10 cc. of milk, *measured accurately*, to a large test tube (50 cc. capacity), add 10 cc. of concentrated HCl, cork tightly, shake thoroughly, and heat in a water bath for about ten minutes, with frequent shaking, until the liquid is of a deep-brown color. The heating must not be continued too long. Cool the tube thoroughly in a stream of water, add about 30 cc. of ether, and shake vigorously. Allow to stand until the ether layer, which contains the fat, has separated out. Transfer as much as possible of the ether layer, without disturbing the other layer, to a weighed flask (this may conveniently be done by closing the test tube with a cork provided with small glass tubes similar to a wash bottle, the larger tube adapted to slide up and down in the cork and turned up slightly at the bottom. When the ether layer is ready to be transferred to the flask, the sliding tube is arranged so that it terminates just above the division of the two layers, and the ether is then blown out into the weighed flask). Add 10 cc. of ether to the test tube and shake again. Transfer this ether layer to the weighed flask. In the same manner make two more

extractions and transfer the ether layer to the weighed flask as before. Evaporate off the ether in the weighed flask, dry at the temperature of boiling water until *free of ether*, cool in desiccator, and weigh. Make duplicate determinations. Calculate and report the percentage of fat in the sample.

Questions

1. Name and describe two other methods for the determination of fat in milk. 2. If the sample of milk is slightly churned, how would you proceed to determine the fat content?

Experiment No. 55

DETERMINATION OF TOTAL PROTEIN (CASEIN AND ALBUMIN) IN MILK

Measure out, with a burette or pipette, two 5 cc. portions of milk into Kjeldahl flasks, and proceed with the digestion, distillation, and titration as in Experiment No. 19. Multiply the percentage of nitrogen by 6.38 to obtain the percentage of total protein in milk. Calculate and report the percentage of total protein in the sample.

Question

How was the factor 6.38 obtained?

Experiment No. 56

DETERMINATION OF CASEIN IN MILK

Measure out, with a burette or pipette, two 10 cc. portions of milk into beakers, dilute with distilled water to a volume of 100 cc., heat to a temperature of 40° to 42° C., and add at once 1.5 cc. of an approximate 10 per cent

acetic acid solution. Stir with a rubber-tipped glass rod, or policeman, and let stand for about five minutes, then decant on filter, wash thoroughly with cold water by decantation (pouring the washings through the filter paper each time), and transfer precipitate *completely* to filter. The filtrate should be clear or very nearly so. If it is not clear when it is first run through, it should again be filtered through the same filter and washed as before. Transfer the filter paper and contents to Kjeldahl flasks and proceed with the digestion, distillation, and titration as in Experiment No. 19. Multiply the percentage of nitrogen by 6.38 to obtain the percentage of casein in the milk. Calculate and report the percentage of casein in the sample.

Questions

1. What is the federal standard for milk? the state standard?
2. What is the composition of cow's milk?
3. How does it differ from human milk?
4. How is cow's milk modified for infants?
5. What facts shown by an analysis would influence you to believe that a sample of milk had been skimmed? watered? skimmed and watered?
6. To what is the acidity of milk due?
7. What is the composition of Cheddar cheese?
8. Give the commercial uses of casein.

Reference

LEACH. Food Inspection and Analysis (3d ed.), p. 160. 1914.

NOTE. Casein may be determined by volumetric methods: Walker, *Journal of Industrial and Engineering Chemistry*, Vol. VI, pp. 131, 356 (1914); Hart, *Journal of Biological Chemistry*, Vol. VI, p. 445 (1909); Van Slyke and Bosworth, *Technical Bulletin No. 10* (1909), New York (Geneva) Experiment Station.

PART VII

A BRIEF SANITARY EXAMINATION OF WATER

Experiment No. 57

DETERMINATION OF TOTAL SOLIDS IN WATER

Evaporate 200 cc. of water in a small evaporating dish on a water bath (a portion of the total volume can be evaporated, then more added until the total amount is evaporated). Dry at $105^{\circ}\text{C}.$ in an oven until weight is constant, cool in desiccator, and weigh. Calculate and report parts of total solids per million parts of water, also in terms of grains per gallon.

Qualitative Analysis

Test the residue for phosphates, chlorides, sulphates, iron, aluminium, magnesium, and calcium.

Experiment No. 58

DETERMINATION OF CHLORINE AS CHLORIDES IN WATER

Measure out, with a pipette or burette, 50 cc. of water into each of two small beakers or evaporating dishes. Add from three to four drops of potassium chromate solution (10 per cent) as an indicator, coloring the contents of each beaker exactly alike. Place the beakers on a white surface. Titrate the water in the beakers with standard

silver nitrate (AgNO_3) solution. Add one drop at a time, and continue the titration with frequent agitation until the water shows the first tinge of red. Make duplicate determinations. If convenient, the titration should be performed under a yellow light or by wearing yellow-colored goggles. Calculate and report parts of chlorine and its equivalent in NaCl per million parts of water.

Questions

1. If the water contains over 5 parts of chlorine per 100,000 parts of water, what is suspected? Discuss. 2. What is the chlorine content of sea water? 3. What is the sanitary significance of the chlorine content of water? 4. Write equations illustrating all reactions. 5. State the relative solubilities of silver chloride and silver chromate.

Experiment No. 59

DETECTION OF FREE AMMONIA IN WATER

To 25 cc. of water in a tall test tube add 5 cc. of Nessler's reagent * and note the color. Only a faint yellow tinge should be visible. A deeper color or turbidity generally indicates animal contamination. Compare the treated sample with the untreated sample in a similar tube. The experiment can be made quantitatively by comparing the color of the sample with different samples of distilled water containing known amounts of ammonium chloride (NH_4Cl).

Questions

1. Of what does Nessler's reagent consist? 2. Define free and albuminoid ammonia. 3. How is albuminoid ammonia determined? 4. Discuss the significance of free and albuminoid ammonia in water.

* Prepared according to directions on page 92.

Experiment No. 60

DETECTION OF NITRITES IN WATER

Into a large test tube place a drop of HCl , 2 cc. sulphanilic acid* and equal volumes of naphthylamine hydrochloride,* and 50 cc. of the water under examination. If a red color is produced immediately or within twenty minutes, the presence of nitrites is assured. As a rule nitrites are not found in good water. Any water containing nitrites should be suspected of being contaminated. Why? The test tube should be corked to avoid contamination from laboratory atmosphere.

Experiment No. 61

DETECTION OF NITRATES IN WATER

Evaporate 100 cc. of the sample to dryness in an evaporating dish over a water bath. Treat with 1 cc. of phenolsulphonic acid,* stirring thoroughly. Add 10 cc. of distilled water and half as much NH_4OH . In the presence of nitrates the characteristic color (yellow) of the ammonia salt of nitrophenol-sulphonic acid is formed. Nitrates are present in almost all natural waters. Why?

* Prepared according to directions on page 93 or by a method proposed by Chamot, Pratt, and Redfield in an article entitled "A Study on the Phenolsulphuric Acid Method for the Determination of Nitrates in Water" (a modified phenolsulphuric acid method), *Journal of American Chemical Society*, Vol. XXXIII, No. 3 (1911). The reagent when prepared by the method recommended by Chamot, Pratt, and Redfield consists of the diacid with only traces of monoacids. This method is especially desirable when the reagent is to be used for quantitative work, as the method of preparation given in this manual yields a mixed product.

Experiment No. 62

DETERMINATION OF ABSORBED OXYGEN IN WATER

Place 100 cc. of water in a beaker and add 10 drops of H_2SO_4 . Warm gently and add the standard solution (KMnO_4), drop by drop (stirring constantly). As soon as the first tinge of pink appears, warm the beaker again and notice if the color is permanent. The first tinge of permanent pink denotes the end of the operation. The test should be limited to about fifteen minutes.

This determination gives reliable information concerning the amount of organic contamination, but does not distinguish between that of vegetable and animal origin. If more than one grain per gallon is absorbed, the water is probably polluted.

Experiment No. 63

DETERMINATION OF TEMPORARY HARDNESS OR ALKALINITY OF WATER

Titrate 100 cc. of water with your standard solution of HCl , using methyl orange or erythrosin and chloroform as indicator. Calculate and report results in parts of calcium carbonate (CaCO_3) per 100,000 parts of water, giving the so-called degrees of hardness. If sodium or potassium carbonates are present, they will also react alkaline, but a correction for this error can be obtained by determining permanent hardness.

Reference

OLSEN. Quantitative Chemical Analysis.

Questions

1. What is hard water? 2. Define permanent hardness and temporary hardness. 3. Give another way in which the hardness of water may be determined. 4. Give the essential determinations on water for the following purposes: (1) drinking, (2) boiler, (3) irrigation. 5. State the benefits derived from each. 6. Discuss the correction of undesirable properties of waters used for different purposes.

PART VIII

APPENDIX

BOOKS OF REFERENCE

- AIKMAN, C. M. Milk, its Nature and Composition.
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- INGLE, H. Manual of Agricultural Chemistry.
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- OLSEN, J. C. Quantitative Chemical Analysis (3d ed.).
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 TALBOT, H. P. Quantitative Chemical Analysis.
 TREADWELL-HALL. Analytical Chemistry. Vol. II, Quantitative Analysis.
 VAN SLYKE, L. L. Modern Methods of testing Milk and Milk Products. 1913.
 WILEY, H. W. Foods and their Adulteration. 1913.
 WILEY, H. W. Principles and Practice of Agricultural Analysis. Vol. I, "Soils" (1906); Vol. II, "Fertilizers and Insecticides" (1908); Vol. III, "Agricultural Products" (1914).

TABLES OF WEIGHTS

Metric System

Milligram =	0.0154 grain
Gram =	15.4323 grains
Gram =	0.03527 ounce avoirdupois
Gram =	0.0321 ounce troy
Kilogram =	2.2046 pounds avoirdupois
Kilogram =	2.6792 pounds troy

Avoirdupois

Long ton =	2240 pounds =	1016.047 kilograms
Short ton =	2000 pounds =	907.184 kilograms
Pound =	16 ounces =	7000 grains = 453.5924 grams
Ounce =	437.5 grains =	28.3495 grams
Grain =	64.798 milligrams =	0.06479

Troy

Pound =	12 ounces =	5760 grains =	373.241 grams
Ounce =	20 pennyweights =	480 grains =	31.103 grams
Pennyweight =	24 grains =	1.555 grams	
Grain =	64.7989 milligrams =	0.06479 gram	

Troy (pharmacy)

Ounce = 8 drams = 480 grains = 31.1034 grams

Dram = 3 scruples = 60 grains = 3.8879 grams

Scruple = 20 grains = 1.295 grams

TABLES OF MEASURES

Length

Millimeter = 0.039 inch

Centimeter = 0.393 inch

Decimeter = 3.937 inches

Meter = 39.37 inches

Meter = 3.280 feet

Meter = 1.0936 yards

Inch = 2.540 centimeters

Foot (12 inches) = 3.0480 decimeters

Yard (3 feet) = 0.914 meter

Mile (1760 yards) = 5280 feet

Mile (1.609347 kilometers) = 1609.347 meters

Surface

Square millimeter = 0.00155 square inch

Square centimeter = 0.1549 square inch

Square decimeter = 15.499 square inches

Square decimeter = 0.1076 square foot

Square meter = 1549.997 square inches

Square meter (10.764 square feet) = 1.195 square yards

Volume

Gallon (U.S.) = 231 cubic inches

Gallon (U.S.) = 3.785 liters

Quart (U.S.) = 0.946 liter

Pint (U.S.) = 0.473 liter

Liter (U.S.) = 2.113 pints (U.S.)

= 1.0566 quarts (U.S.)

= 0.264 gallon (U.S.)

Cubic meter = 1.307 cubic yards

= 35.314 cubic feet

An imperial gallon (English) = 4.545 liters

= 277.410 cubic inches (U.S.)

STRENGTH OF HCl SOLUTION AT DIFFERENT
DENSITIES, 15° C.

SPECIFIC GRAVITY	PER CENT OF HCl	GRAMS HCl IN 100 cc.	SPECIFIC GRAVITY	PER CENT OF HCl	GRAMS HCl IN 100 cc.
1.095	19.06	20.9	1.150	29.57	34.0
1.100	20.01	22.0	1.155	30.55	35.3
1.105	20.97	23.2	1.160	31.52	36.6
1.110	21.92	24.3	1.165	32.49	37.9
1.115	22.86	25.5	1.170	33.46	39.2
1.120	23.82	26.7	1.175	34.42	40.4
1.125	24.78	27.8	1.180	35.39	41.8
1.130	25.75	29.1	1.185	36.31	43.0
1.135	26.70	30.3	1.190	37.23	44.3
1.140	27.66	31.5	1.195	38.16	45.6
1.145	28.61	32.8	1.200	39.11	46.9

STRENGTH OF H₂SO₄ SOLUTION AT DIFFERENT
DENSITIES, 15° C.

SPECIFIC GRAVITY	PER CENT OF H ₂ SO ₄	GRAMS H ₂ SO ₄ IN 100 cc.	SPECIFIC GRAVITY	PER CENT OF H ₂ SO ₄	GRAMS H ₂ SO ₄ IN 100 cc.
1.700	77.17	131.2	1.790	85.70	153.4
1.710	78.04	133.4	1.800	86.90	156.4
1.720	78.92	135.7	1.810	88.30	159.8
1.730	79.80	138.1	1.820	90.02	163.9
1.740	80.68	140.4	1.825	91.00	166.1
1.750	81.56	142.7	1.830	92.10	168.5
1.760	82.44	145.1	1.835	93.43	171.3
1.770	83.32	147.5	1.840	95.60	175.9
1.780	84.50	150.4			

STRENGTH OF HNO_3 SOLUTION AT DIFFERENT
DENSITIES, 15°C .

SPECIFIC GRAVITY	PER CENT OF HNO_3	GRAMS HNO_3 IN 100 CC.	SPECIFIC GRAVITY	PER CENT OF HNO_3	GRAMS HNO_3 IN 100 CC.
1.20	32.36	38.8	1.37	59.39	81.4
1.21	33.82	40.9	1.38	61.27	84.6
1.22	35.28	43.0	1.39	63.23	87.9
1.23	36.78	45.2	1.40	65.30	91.4
1.24	38.29	47.5	1.41	67.50	95.2
1.25	39.82	49.8	1.42	69.80	99.1
1.26	41.34	52.1	1.43	72.17	103.2
1.27	42.87	54.4	1.44	74.68	107.5
1.28	44.41	56.8	1.45	77.28	112.1
1.29	45.95	59.3	1.46	79.98	116.8
1.30	47.49	61.7	1.47	82.90	121.9
1.31	49.07	64.3	1.48	86.05	127.4
1.32	50.71	66.0	1.49	89.60	133.5
1.33	52.37	69.7	1.50	94.09	141.1
1.34	54.07	72.5	1.51	98.10	148.1
1.35	55.79	75.3	1.52	99.67	151.5
1.36	57.57	78.3			

STRENGTH OF NH_4OH SOLUTION AT DIFFERENT
DENSITIES, 15°C .

SPECIFIC GRAVITY	PER CENT OF NH_3	GRAMS NH_3 IN 100 CC.	SPECIFIC GRAVITY	PER CENT OF NH_3	GRAMS NH_3 IN 100 CC.
.936	16.82	15.74	.916	23.03	21.09
.934	17.42	16.27	.914	23.68	21.63
.932	18.03	16.81	.912	24.33	22.19
.930	18.64	17.34	.910	24.99	22.74
.928	19.25	17.86	.908	25.65	23.29
.926	19.87	18.42	.906	26.31	23.83
.924	20.49	18.93	.904	26.98	24.39
.922	21.12	19.47	.902	27.65	24.94
.920	21.75	20.01	.900	28.33	25.50
.918	22.39	20.56	.898	29.01	26.05

STRENGTH OF NaOH SOLUTION AT DIFFERENT
DENSITIES, 15° C.

SPECIFIC GRAVITY	BAUMÉ	PER CENT OF NaOH	GRAMS NaOH IN 100 CC.
1.075	10	6.55	7.0
1.083	11	7.31	7.9
1.091	12	8.00	8.7
1.100	13	8.68	9.5
1.108	14	9.42	10.4
1.116	15	10.06	11.2
1.125	16	10.97	12.3
1.134	17	11.84	13.4
1.142	18	12.64	14.4
1.152	19	13.55	15.6
1.162	20	14.37	16.7
1.171	21	15.13	17.7
1.180	22	15.91	18.8
1.190	23	16.77	20.0
1.200	24	17.67	21.2
1.210	25	18.58	22.5
1.220	26	19.58	23.9
1.231	27	20.59	25.3
1.241	28	21.42	26.6
1.252	29	22.64	28.3
1.263	30	23.67	29.9
1.274	31	24.81	31.6
1.285	32	25.80	33.2
1.297	33	26.83	34.8
1.308	34	27.80	36.4
1.320	35	28.83	38.1
1.332	36	29.93	39.9
1.345	37	31.22	42.0
1.357	38	32.47	44.1
1.370	39	33.69	46.2
1.383	40	34.96	48.3
1.397	41	36.25	50.6
1.410	42	37.47	52.8
1.424	43	38.80	55.3
1.438	44	39.99	57.5
1.453	45	41.41	60.2

SOLUBILITIES IN WATER

All the chlorides are soluble except those of *silver*, *lead*, and *mercurous mercury*.

All the sulphates are soluble except those of *strontium*, *barium*, and *lead*.

All the carbonates and phosphates are insoluble except those of *sodium*, *potassium*, and *ammonium*.

All the hydroxides are insoluble except those of *sodium*, *potassium*, *ammonium*, *calcium*, *strontium*, and *barium*.

All nitrates, acetates, and chlorates are soluble.

DIRECTIONS FOR PREPARATION OF REAGENTS

INDICATORS FOR VOLUMETRIC ANALYSIS

Litmus. Boil 1 g. of purified powdered litmus with 60 cc. of distilled water, filter, and divide filtrate into two equal portions. To one portion add dilute H_2SO_4 , drop by drop, until the solution is just acid. Mix the two portions and keep in a glass-stoppered bottle.

Cochineal. Digest 1 g. of crushed cochineal dregs in 100 cc. of 25 per cent alcohol (be sure the alcohol is neutral) and filter.

Methyl Orange. Dissolve 1/10 g. in 100 cc. of distilled water.

Methyl Red. Dissolve 1/10 g. in 100 cc. of distilled water.

Phenolphthalein. Dissolve 1 g. in 100 cc. of 50 per cent alcohol.

Corallin. Saturate alcohol (that has previously been made neutral) with corallin.

I. *Solutions for Quantitative Analysis*

Asbestos for Gooch Crucible. Select a good grade of asbestos with long fibers. The asbestos should be separated until the fibers are about one-fourth inch long, then digested with concentrated HCl for twelve hours, filtered, and washed with distilled water until free from chlorides. Transfer the asbestos to a bottle containing distilled water

Silver Nitrate Solution. Dissolve 16.994 g. of silver nitrate (AgNO_3) crystals in 1000 cc. of distilled water (free from chlorides) and place in a dark-colored bottle away from the sunlight.

Ammonium Oxalate Solution. Dissolve 42 g. of ammonium oxalate ($(\text{NH}_2)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$) in 1000 cc. of distilled water.

II. *Solutions used in the Analysis of Feedstuff*

Saturated Solution of Sodium Hydroxide. Dissolve about 1000 g. of NaOH in 1 liter of water, cool, and pour into a bottle (crude NaOH may be used if the impurities are allowed to settle and the clear solution drawn off for use).

Anhydrous, Alcohol-Free Ether. Let the ether stand in contact with calcium chloride (CaCl_2 , 50 g. to about 500 cc.) until next laboratory period, and distill, using a distilling tube. Place the distilled ether in a *dry* glass-stoppered bottle over metallic sodium free from oil. For a short time leave the bottle loosely stoppered so that the hydrogen evolved may escape. The ether should be *filtered*, or the clear solution should be drawn off *without disturbing the residue*.

III. *Solutions used in the Analysis of Soils*

Ammonium Oxalate Solution. Prepared as in I.

Sodium Ammonium Hydrogen Phosphate (Microcosmic Salt). Dissolve 100 g. of sodium ammonium hydrogen phosphate ($\text{NaNH}_4\text{HPO}_4 \cdot 4\text{H}_2\text{O}$) in 1 liter of distilled water.

Ammonium Molybdate Solution.* Dissolve 100 g. of molybdic acid in 144 cc. of ammonium hydroxide (sp. gr. 0.90) and 271 cc. of water; slowly, and with constant stirring, pour the solution thus obtained into 489 cc. of nitric acid (sp. gr. 1.42) and 1148 cc. of water. Keep the mixture in a warm place for

* *Bulletin 107* (Revised), Bureau of Chemistry, United States Department of Agriculture.

several days or until a portion heated to 40° C. deposits no yellow precipitate of ammonium phosphomolybdate. Decant the solution from any sediment and preserve in a glass-stoppered bottle.

Ammonium Carbonate Solution. Add 250 cc. of ammonium hydroxide (sp. gr. 0.90) to 250 g. ammonium carbonate ($(\text{NH}_4)_2\text{CO}_3$) and make up to a liter.

Saturated Solution of Sodium Hydroxide. Prepared as in I.

Saturated Solution of Barium Hydroxide. Dissolve about 50 g. of barium hydroxide in 1 liter of water.

Platinum Solution. Dissolve 172.8 g. PtCl_4 in 1 liter of water. (1 cc. of the solution contains .1 g. of platinum, equivalent to .21 g. H_2PtCl_6 .)

Solution of Oxalic Acid. Dissolve 3.2 g. of oxalic acid ($\text{C}_2\text{H}_2\text{O}_4 \cdot 2 \text{H}_2\text{O}$) in 500 cc. of distilled water containing 25 cc. of concentrated C. P. H_2SO_4 . Measure out, with a pipette or burette, two 25 cc. portions into beakers, add 2 cc. of H_2SO_4 (1:1), heat to 60° C., and titrate with the standard KMnO_4 solution. Note the volume of KMnO_4 solution required to oxidize completely 1 cc. of the oxalic acid solution.

Ammonium Chloride Solution saturated with Potassium Chloroplatinate. Dissolve 100 g. of ammonium chloride in 500 cc. of water, add from 5 to 10 g. of pulverized potassium chloroplatinate (potassium platinic chloride), and shake at intervals for six or eight hours. Allow the mixture to settle, filter, and use the clear filtrate for the potash determination. The residue may be used for the preparation of a fresh supply.

Cobaltinitrite Reagent.* Dissolve 220 g. of sodium nitrite in 400 cc. of water and 113 g. of cobalt acetate in 300 cc. of water, and add 100 cc. of glacial acetic acid. The two solutions are mixed and gently warmed, NO_2 is evolved, and the solution becomes dark colored. The NO_2 is best evacuated from the bottle by a water pump and the liquid left overnight,

* F. Sutton, Volumetric Analysis (9th ed.), p. 62.

during which a yellow precipitate settles. The solution is then filtered and diluted with water to 1 liter.

Alcohol Solution. Prepare the alcohol solution containing 80 per cent alcohol (sp. gr. .8639 at 15° C.).

Asbestos for Gooch Crucible. Prepared as in I.

IV. *Solutions used in the Analysis of Fertilizers*

Ammonium Molybdate Solution. Prepared as in III.

Ammonium Oxalate Solution. Prepared as in I.

Ammonium Chloride Solution saturated with Potassium Chloroplatinate. Prepared as in III.

Asbestos for Gooch Crucible. Prepared as in I.

Alcohol Solution (80%). Prepared as in III.

Cobaltinitrite Reagent. Prepared as in III.

Platinum Solution. Prepared as in III.

Magnesia Mixture. Weigh out 11 g. of recently ignited calcined magnesia and dissolve in dilute hydrochloric acid, avoiding an excess. Add a little excess of magnesia and boil to precipitate iron, alumina, and phosphoric acid. Filter, add 140 g. ammonium chloride and 130.5 g. of ammonium hydroxide (sp. gr. .9), and dilute to 1 liter. Instead of the calcined magnesia, 55 g. of crystallized magnesium chloride ($\text{MgCl}_2 \cdot 6 \text{H}_2\text{O}$) may be used.

Salicylic Acid Solution. Add 1 g. salicylic acid to 30 cc. H_2SO_4 . Shake until thoroughly mixed, and allow it to stand from five to ten minutes, with frequent shakings.

Ammonium Citrate Solution.* Dissolve 370 g. of commercial citric acid in 1500 cc. of water; nearly neutralize with

* Hall, *Journal of Industrial and Engineering Chemistry*, Vol. III (1911), p. 559; Rudnick, *Journal of Industrial and Engineering Chemistry*, Vol. V (1913), pp. 12, 998; E. D. Eastman and J. H. Hildebrand, *Journal of Industrial and Engineering Chemistry*, Vol. VI (1914), p. 578; Hall, *Journal of American Chemical Society*, Vol. XXXVII (January, 1915), p. 208. *Bulletin 107* (Revised), Bureau of Chemistry, United States Department of Agriculture.

commercial ammonium hydroxide; cool; add ammonium hydroxide until exactly neutral (testing with saturated alcoholic solution of corallin); and dilute to a volume of 2 liters. Determine the specific gravity, which should be 1.09 at 20° C.

V. *Solutions used in the Analysis of Insecticides*

Alcohol Solution (95% C_2H_5OH by Volume). Prepare a solution of alcohol whose specific gravity is .8164 at 15° C.

Potassium Ferrocyanide Solution. Dissolve 84 g. of potassium ferrocyanide ($K_4Fe(Cn)_6$) in 1 liter of distilled water.

VI. *Solutions used in the Analysis of Milk*

Anhydrous, Alcohol-Free Ether. Prepared as in II.

Saturated Sodium Hydroxide Solution. Prepared as in II.

VII. *Solutions used in a Brief Sanitary Examination of Water*

Potassium Chromate Solution (Indicator). Dissolve 10 g. of potassium chromate (K_2CrO_4) in 100 cc. of distilled water.

Nessler's Reagent.* Dissolve 62.5 g. of potassium iodide in about 250 cc. of distilled water, set aside a few cubic centimeters, and add gradually to the larger part a cold saturated solution of mercuric chloride (of which about 500 cc. will be required) until the mercuric iodide precipitated ceases to redissolve on stirring. When a permanent precipitate is retained, restore the reserved potassium iodide so as to redissolve it, and continue adding mercuric chloride very gradually until a slight precipitate remains undissolved. (The small quantity of potassium iodide is set aside merely to enable the mixture to be made rapidly without danger of adding an excess of mercury.) Next dissolve 150 g. of potassium hydroxide in 150 cc. distilled water, allow the solution to cool, add it gradually to the above solution, and make up with distilled water to 1 liter.

* F. Sutton, Volumetric Analysis (10th ed.), p. 437.

On standing, a brown precipitate is deposited and the solution becomes clear and of a pale greenish-yellow color. It is ready for use as soon as it is completely clear, and should be decanted into a smaller bottle as required. The reagent improves on keeping.

Sulphanilic Solution. Dissolve .8 g. of the acid in 100 cc. of distilled water, heating if necessary.

Naphthylamine Hydrochloride Solution. Dissolve .8 g. of the salt in 100 cc. of hot distilled water to which 1 cc. of HCl has been added. Filter through bone black, or add bone black to the solution, and decant as needed. Keep away from the light.

Phenolsulphonic Acid. Mix 30 g. of phenol with 210 cc. or 370 g. of concentrated H_2SO_4 in a flask. Place the flask in a water bath so that the surface of the liquid in the flask will be below the water. Heat for six hours at the temperature of boiling water.

APPARATUS FOR DESK EQUIPMENT

4 beakers: two 200 cc.; two 350 cc.	2 flasks, measuring: one 250 cc.;
5 bottles: two 500 cc.; two 1000 cc.;	one 500 cc.
one 250 cc. (wide-mouthed).	1 flask, filtering.
2 Bunsen burners with 4 ft. of	5 funnels: four 50 mm.; one
rubber tubing.	Gooch.
2 burettes, complete.	1 funnel holder, wood.
1 burette holder.	Matches, safety, 1 box.
1 condenser with clamp.	2 pipettes: one 25 cc.; one 50 cc.
1 crucible tongs.	2 rings, iron, 3-inch.
2 crucibles, porcelain, No. 7, with	4 rods, glass.
covers.	1 stand, iron.
2 crucibles, Gooch.	6 test tubes, 10 cm.
1 cylinder, graduated, 50 cc.	1 test-tube brush.
1 desiccator, complete.	1 test-tube rack.
2 evaporating dishes, No. 4.	1 thermometer, 100°C .
25 filter papers, 9 cm.	Towel or one-half yard absorption
1 flask, 500 cc. with rubber stop-	cloth.
per for wash bottle.	2 triangles, pipetem.
4 flasks, Erlenmeyer: two 250 cc.;	Tubing, glass, for wash bottle.
two 500 cc.	4 watch glasses: one 35 mm.; one
2 flasks, Kjeldahl digestion: 800 cc.	50 mm.; one 62 mm.; one 80 mm.

INTERNATIONAL ATOMIC WEIGHTS (1916)

Abridged Table

ELEMENT	SYMBOL	ATOMIC WEIGHT	ELEMENT	SYMBOL	ATOMIC WEIGHT
Aluminium	Al	27.10	Lithium	Li	6.94
Antimony	Sb	120.20	Magnesium	Mg	24.32
Arsenic	As	74.96	Manganese	Mn	54.93
Barium	Ba	137.37	Mercury	Hg	200.6
Bismuth	Bi	208.00	Molybdenum	Mo	96.0
Boron	B	11.00	Nickel	Ni	58.68
Bromine	Br	79.92	Nitrogen	N	14.01
Calcium	Ca	40.07	Oxygen	O	16.0
Carbon	C	12.00	Phosphorus	P	31.04
Chlorine	Cl	35.46	Platinum	Pt	195.2
Chromium	Cr	52.00	Potassium	K	39.10
Cobalt	Co	58.97	Silicon	Si	28.30
Copper	Cu	63.57	Silver	Ag	107.88
Fluorine	F	19.00	Sodium	Na	23.00
Gold	Au	197.20	Strontium	Sr	87.63
Hydrogen	H	1.008	Sulphur	S	32.06
Iodine	I	126.92	Tin	Sn	118.70
Iron	Fe	55.84	Uranium	U	238.20
Lead	Pb	207.20	Zinc	Zn	65.37

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