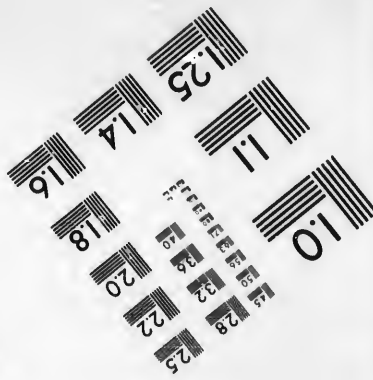
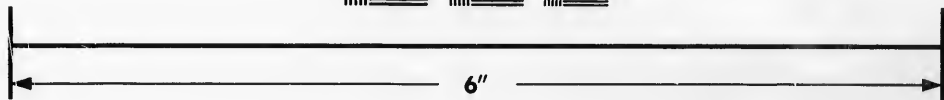
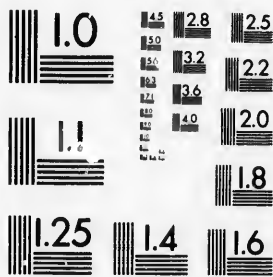


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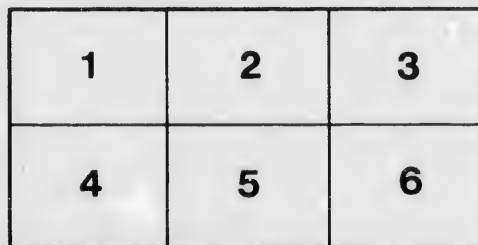
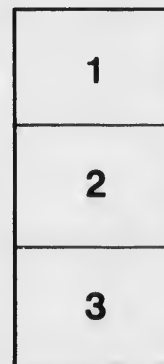
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A  
LABORATORY HANDBOOK  
OF  
Experimental Chemistry

FOR THE USE OF  
DENTAL STUDENTS.

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COMPILED BY

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TORONTO, ONTARIO.

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TORONTO:  
MONETARY TIMES PRINTING COMPANY, LIMITED,  
1897.





## PREFACE.

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This book, as its title indicates, is intended for the use of dental students in the chemical laboratory. As such it is designed to satisfy the hitherto unsatisfied need for such. The course in chemistry which the dental student must cover is rather peculiar and one which is not found in the ordinary text books. His working knowledge of chemistry must be essentially practical. One of the objects of his course is to acquaint himself thoroughly with the operations which he must perform in his own laboratory. To this end the variety of subjects covered in this course is rather extensive. The elementary study of the principal non-metals is necessary as a foundation to all further study of chemistry. Then it is important that a working knowledge of qualitative analysis both in a dry and wet way, should be obtained. Metals, such as are found in the dental laboratory, also must come in for examination and treatment. Many organic substances, including the alkaloids, must not be overlooked. Physiological chemistry also has a claim. Such a variety of work does not exist in any one work suitable for use in the laboratory. Hence the appearance of the present course. As a laboratory hand book it is not of course complete in itself, but must be supplemented by lectures and also by independent reading on the part of the student. By this means a knowledge of the science as well as the art of chemistry will be gained.

In conclusion the compiler would say that he claims nothing to be original but the arrangement. Many sources of information have been made use of, which are as varied in character as the course itself.

W. P. C.

TORONTO, Oct. 1st, 1897.



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## TO THE STUDENT.

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1. Full notes should be made of every experiment performed. These should consist of a full description of the various phenomena noticed, and also of a statement of any inference to be derived from such phenomena. As this is not in any sense a text book, outside reading will be necessary. The result of this should appear in the form of explanations of experiments. The book may be called in any day for examination by the Demonstrator. Use formulæ and equations where possible.
2. Cultivate the art of using small quantities in working. By this means greater nicety of operation will be secured.
3. Keep all apparatus in a clean, neat state. Do not allow acids to remain on the outside of your bottles. Let the drawers and lockers be kept in a tidy condition.
4. Nothing should be put into reagent bottles, not even stirring rods or test paper. Never pour back into its bottle a reagent once removed.
5. Never allow solid materials to be put into the sink. The earthenware bowls are for that purpose. Always allow the water to run after acids have been poured down the escape pipe.
6. Never lay the stoppers of reagent bottles down. Hold them between the first and second fingers.
7. When heating a substance in a test tube always hold the mouth of the tube away from you.
8. Unless otherwise stated every student is expected to work independently.
9. Reagents at the Demonstrator's table are for general use, and must not be taken to the individual desks.
10. Always read the directions before you begin an experiment. Be sure you understand what you are to do before you start.

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## SECTION I.

### 1.—HYDROGEN. Symbol, H. At. Wt., 1.

**PREPARATION.**—Take a wide mouthed bottle and fit it with a good cork perforated by two glass tubes, one of which passes nearly to the bottom of the bottle, and has on its upper end a funnel-like expansion; the other tube merely passes through the cork and is bent at right angles, and has a rubber tube attached to it for conveying the gas to a "pneumatic trough." Place some clippings of zinc in the bottle, fill it about one-third full of water, and then pour down the funnel tube about 10 cc. of Sulphuric Acid. The gas begins to form quickly, and is collected in test tubes previously filled with water and kept mouth downward in the water. *The first portion must be rejected, as air and Hydrogen form an explosive mixture.* It is therefore necessary to wait until all the air has been expelled from the flask by the Hydrogen. Before filling all the test tubes, collect one full of the gas over the pneumatic trough, and apply a light to it (holding the mouth downwards); if it burn quietly, you may safely proceed to fill the test tubes, but if a slight explosion occur, you must wait until another test-tube is collected and found to burn quietly. Collect three test tubes of the gas.

Write the equation for the reaction.

What general class of reactions does it represent?

**PROPERTIES.**—Apply a light to one of the test tubes of Hydrogen. What do you observe? What is formed? What property of Hydrogen does it illustrate?

Take the second tube, and keeping its mouth downwards, quickly turn its mouth upwards under the mouth of a similar tube filled with air. Let it remain thus for a few seconds, and then apply a burning taper to the mouth of the upper vessel.

What property of Hydrogen does this illustrate?

Take another jar of Hydrogen, held mouth downwards, and push up into it a lighted taper, supported on a straight wire. The Hydrogen will burn at the mouth of the jar, but the taper will be extinguished. The taper may be withdrawn, re-lighted, and re-extinguished two or three times.

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Does Hydrogen support combustion?

Remove the conducting tube from the Hydrogen flask above described (but without removing the stopper), and substitute for it a straight piece of glass tube of the same diameter, drawn out at the upper end so as to form a jet. After all the air has long ago been expelled, the Hydrogen issuing from the jet may be safely lighted. *Be very careful that all the air has been expelled from the generator before the jet is lighted.* Why?

Notice the color of the flame. Hold a cold porcelain dish over the flame. What is formed?

What are the properties of Hydrogen?

Write the equations for the above experiments.

## 2.—OXYGEN. Symbol. O. At. Wt., 16.

PREPARATION.—Place a small quantity of Mercuric Oxide in a dry test tube, and heat it over a Bunsen lamp. The substance darkens in colour, and a ring of minute globules of Mercury soon forms on the cool part of the tube. Introduce a glowing splinter into the test tube. What happens? Why?

Write the equation representing the above reaction.

Place in a *dry* flask a mixture of 10 grams of Potassic Chlorate and  $2\frac{1}{2}$  grams Manganese Dioxide. Place this on a sand bath, and support the apparatus on a retort stand. Fit the neck of the flask with a stopper and a conducting tube which leads to a pneumatic trough. Heat strongly and carefully, collect over water three beakers and one test tube full of the gas which comes off.

Be sure to remove the delivery tube from the water before the heat is taken away.

PROPERTIES.—Plunge a glowing splinter into a test tube full of the gas. What happens?

Place a few pieces of charcoal, about the size of peas, in a deflagrating spoon; hold this in a lamp flame till the charcoal is just kindled, and then plunge the spoon into a jar of Oxygen. What do you observe? What is formed?

Without allowing any of the gases to escape shake up some lime water in the beaker in which the charcoal has been burned. What is lime water a test for?

Place a few pieces of Sulphur in the deflagrating spoon, heat until the Sulphur is melted and takes fire, and then plunge it into a jar of Oxygen.

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Write the equation for the above reaction. Place some water in the jar, shake, and test with litmus. What has been formed? What is an Anhydride?

Clean the spoon used in the last experiment, and place a piece of Phosphorus on it about the size of a pea. Ignite, and place in a jar of Oxygen. After combustion has ceased, remove the spoon, and pour some water into the jar. Shake up the water with the product of the combustion, and then add some blue litmus solution.

Explain the reactions.

What are the properties of Oxygen?

Pour a little Potassium Hydroxide into a jar of Oxygen. Shake, and if no change in volume takes place, add a small quantity of Pyrogallic Acid. Shake again, and note any change.

Of what practical value is this experiment?

---

### 3.—OZONE. Symbol $O_3$ . Mol. Wt., 48.

Prepare some test paper as follows:—Take a piece of starch about the size of a pea, pulverize and add about 100 cc. of water. Boil to a thin paste; add a few crystals of Potassium Iodide. Dip into this mixture some strips of paper from a scribbler.

In a beaker place two grams of Potassium Permanganate crystals. On these pour some Sulphuric Acid. *Do not heat.* Hold some of the test paper above the mouth of the beaker. What occurs? Explain.

When the gas is coming off freely hang over it, but so as not to touch the liquor, a paper saturated with turpentine. Explain the resulting phenomenon.

Explain the chemical actions of Ozone.

---

### 4.—HYDROGEN DIOXIDE (Hydrogen Peroxide.)—Symbol $H_2O_2$ , Mol. Wt., 34.

To a quantity of dilute  $H_2SO_4$  add some powdered Barium Dioxide,  $BaO_2$ . Filter off the precipitate. What does the *filtrate* contain? Write the equation. Divide the solution into three parts.

Into one drop some litmus solution. Does any change take place?

Into another place a piece of the test paper used for Ozone. What do you observe?

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Make a slightly coloured solution of Potassium Permanganate. To it add some Hydrogen Dioxide solution. Shake the two together and let them stand for some time. Does the Permanganate lose colour?

Explain fully the chemical action of Hydrogen Dioxide. What is meant by nascent state?

— —

**5.—NITROGEN. Symbol N At. Wt., 14.**

Place a small piece of dry Phosphorus on a porcelain crucible lid, and fix this on a flat piece of cork, so that the lid may be floated on the water in the pneumatic trough. Light the Phosphorus, and quickly surround it with a beaker. A portion of the air will first be expelled by the heat, and the Phosphorus will continue to burn as long as any Oxygen remains in the air of the bell-jar; when that is exhausted it will cease to burn. Observe that the water rises inside the bell-jar, replacing the Oxygen, which is no longer gaseous, but has combined with the Phosphorus to form Phosphorus Pentoxide. Wait a few minutes to allow the Pentoxide to be completely dissolved by the water.

What is contained in the beaker?

Plunge a lighted taper into the jar.

What are the properties of Nitrogen? Compare them with those of Oxygen and Hydrogen.

—

**6—NITROGEN MONOXIDE (Nitrous Oxide.) Symbol, N<sub>2</sub>O. Mol. Wt., 44.**

PREPARATION.—Put 25 grams of commercial Ammoniac Nitrate,  $\text{NH}_4\text{NO}_3$ , into an Oxygen generating apparatus, connected with three wash bottles. The first bottle should contain a solution of Ferrous Sulphate, the second, a solution of Caustic Potash, and the third, water. Heat the Nitrate gently and Nitrogen Monoxide will be given off. Collect four beakers of the gas over *warm* water.

If the Nitrate be chemically pure, the wash bottles may be omitted. Write the equation for the above reaction.

Perform the following experiments;

(a) Plunge a lighted taper into the first jar.

(b) Burn a piece of Phosphorus, or Carbon, or Sulphur in the second jar. If burning strongly at first, they continue to burn. Write the equation expressing the reaction,

(c) Explode a mixture of the gas with Hydrogen. Write the equation.

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(d) Place the fourth jar, mouth downward, over *cold* water, and then shake.

Why is the gas collected over warm water ?

What are the properties of Nitrogen Monoxide ?

Why are the wash bottles used ?

#### 7.—NITROGEN DIOXIDE (Nitric Oxide). Symbol, NO. Mol. Wt. 30.

PREPARATION.—Place some copper filings in a Hydrogen generating apparatus, add some warm water, and then pour down the funnel tube some strong Nitric Acid. The gas that first forms should be allowed to escape.

Collect over water four beakers full of the gas and perform the following experiments:—

(a) Allow the contents of the first beaker to escape into the air.

(b) Ignite a piece of Phosphorus very slightly and plunge it into the second beaker.

(c) Allow another piece of Phosphorus to burn strongly, and then place it in the third beaker.

(d) Pour a solution of Ferrous Sulphate,  $\text{FeSO}_4$ , into the fourth beaker full of the gas. Then hold the hand over the beaker's mouth and shake vigorously. Note the two phenomena that occur.

Write all the equations for the above experiments. Enumerate the properties of Nitric Oxide.

#### 8.—NITRIC ACID. Symbol, $\text{HNO}_3$ . Mol. Wt., 63.

PREPARATION.—Put into a tabulated glass retort 30 grams of powdered Nitrate of Potash,  $\text{KNO}_3$ , and an equal weight of strong Sulphuric Acid,  $\text{H}_2\text{SO}_4$ . Place the end of the retort in a flask which is made to float on a basin of water. Apply heat to the retort. Soon a yellowish coloured liquor distils over and is collected in the cool flask. Write the equation.

Nitric Acid an oxidizing agent :

(a) Place a small piece of Phosphorus in a saucer, then drop on it a little of the acid.

(b) On powdered pieces of glowing charcoal pour a little Nitric Acid.

Tests for Nitric Acid :

(a) Add a few drops of the Nitric Acid prepared as above to a solution of Indigo contained in a test-tube. Notice that the blue colour speedily disappears.

(b) Place a few bits of copper turnings in a test-tube, cover

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them with water, and add a little Nitric Acid. The Copper soon begins to dissolve, forming a blue solution, and at the same time brown vapours fill the test-tube.

Explain the above reaction.

(c) Dissolve a few crystals of Ferrous Sulphate,  $\text{FeSO}_4$ , in water in a test-tube. Add a few drops of Sulphuric Acid and allow the whole to cool. Then turn the test-tube sideways and gently pour Nitric Acid or a nitrate in solution down its side. The phenomenon which results will always enable us to recognize Nitric Acid or a nitrate.

To the formation of what substance is the black ring due?

### 9.—AMMONIA. Symbol $\text{NH}_3$ Mol. Wt., 17.

PREPARATION.—Mix ten grams of Ammonic Chloride and the same weight of Quicklime. Place the mixture in a flask provided with a long delivery tube passing straight upwards. Heat gently. When the gas begins to come off invert a test tube over the end of the delivery tube. Is Ammonia lighter or heavier than air? How then may it be collected?

PROPERTIES :—(a) Pass a lighted taper up into the test tube full of gas.

(b) Pass some of the gas into reddened litmus. Upon the result of this, devise a means of knowing when a bottle is full of this gas.

(c) Moisten a glass rod with Hydrochloric Acid, and then bring it near the end of the delivery tube. Do the same with other acids.

(d) Fill a test tube with Ammonia, and invert it over a beaker of cold water coloured with red litmus.

Account for the resulting phenomenon.

TESTS.—Will any of the above actions answer as tests for Ammonia?

When present in minute quantities, as it frequently is in drinking water, Ammonia is best detected by what is known as Nessler's test: "To a solution of Potassic Iodide add solution of Mercuric Chloride until the precipitate formed just ceases to be re-dissolved, then, add an equal volume solution of Caustic Potash, and allow the whole to stand until clear. A few drops of this solution will give a yellowish-brown precipitate, with even the slightest trace of Ammonia,"

What are the properties of Ammonia?

Explain by means of an equation the method of its preparation.

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## 10.—CHLORINE. Symbol, Cl. At. Wt., 35.5.

PREPARATION.—1. Into a test-tube put one part of Manganese Dioxide, two parts of salt, and three of Sulphuric Acid. Fit the test-tube with a cork and delivery tube. Heat gently and pass the gas that comes off into separate solutions of litmus and indigo. Cautiously smell the gas. Note its colour.

2. To prepare the gas on a larger scale, take a Florence flask and place in it about 20 grams of Manganese Dioxide and 100 ccs. of strong Hydrochloric Acid. The flask should be fitted with a delivery tube bent over so as to reach almost to the bottom of a collecting beaker. The beaker should set on the table and should be covered with a piece of cardboard through which the delivery tube passes. Why can not Chlorine be collected over water? Apply a very gentle heat. Fill several jars, taking care that little or none of the gas escapes into the room. Afterwards pass the gas into a flask perfectly full of water; in about ten minutes place this flask aside for future use. Smell the water.

PROPERTIES.—(a) Take the flask full of Chlorine water prepared in the last experiment, and fit it with a cork and tube. The outer end of the tube must be drawn to a fine point. Insert the cork so that there is not a bubble of air left in the flask. Invert the flask and expose to direct sunlight for a day. Then place the flask on the table, remove the cork, and quickly bring a glowing splinter to the mouth of the flask. Test the water in the flask with blue litmus solution. Taste it.

What happens the glowing splinter? Why?

(b) Lower very slowly a lighted taper into a jar of Chlorine. At the same time suspend a piece of blue litmus paper at the mouth of the jar. Smell the gas that is formed during the combustion. How can you account for the action of the taper?

(c) Wet a piece of blotting paper with Oil of Turpentine,  $C_{10}H_{16}$ , and then place it in another jar of the gas.

(d) Take a few pieces of the metal antimony and powder them; then place on a sheet of paper and shake the powder into a jar of Chlorine. What compound is formed in the above experiment?

(e) Place a piece of red cloth, partly moistened and partly dry, in a jar of Chlorine.

Explain fully the bleaching action of Chlorine and compare it with the similar action of Ozone and Hydrogen Dioxide.

Make a list of the principal properties of Chlorine.

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**11.—HYDROCHLORIC ACID. Symbol, HCl. Mol. Wt., 36.5.**

PREPARATION.—In a flask provided with a thistle tube and delivery tube similar to the one used in the preparation of Chlorine, place 30 grams of common salt. Add by degrees 50 grams of strong  $H_2SO_4$ . What is the specific gravity of  $H_2SO_4$ ? After the first evolution of gas has taken place heat gently. Collect by downward displacement of air. Describe the gas which comes off. Write the equation.

PROPERTIES.—(a) Invert a test tube full of the gas over a beaker of water, coloured with red litmus.

(b) Fill a beaker with the gas. Place a piece of Sodium in a deflagrating spoon, heat the Sodium until ignition and lower it into the jar of gas, which must be tightly covered. After the action has ceased quickly bring a lighted taper to the mouth of the beaker. What has been formed? Explain. Dissolve the solid on the spoon. Taste it. What is it? Write all equations.

Test for Hydrochloric Acid or a soluble Chloride.

To a solution of Hydrochloric Acid, or a Chloride, add a few drops of Nitrate of Silver solution.

What is formed? Write the equation.

**12.—IODINE. Symbol, I. At. Wt., 127.**

TEST FOR FREE IODINE.—Powder a piece of starch (about the size of a pea) in a mortar, stir it up with about 25 c.c. of cold water, and then heat the mixture (preferably in an evaporating basin) till it boils. A thin, clear solution of starch is thus obtained; add a portion of it to about a quarter of a litre of water, and then a few drops of one of the solutions of Iodine, and observe the deep blue colour which the liquid assumes. Heat a little of this blue liquid in a test tube, and observe that the colour disappears; allow it to cool, and observe the reappearance of the colour.

How may certain Iodine compounds be tested? Why have substances the power to free Iodine from the Potassium Salt?

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**13.—CARBON.—Symbol. C. At. Wt., 12.**

PROPERTIES.—(a) Place a wet filter paper inside a funnel, and then cover the inside of the filter paper with a thick coating of animal charcoal, or bone black. Now filter through the paper a wine-glass full of ale or porter.

(b) Place a piece of charcoal in a test-tube and then pour upon it a little strong Sulphuric Acid. Observe whether the charcoal changes in any way. Try whether an alkali will produce any change in the charcoal.

(c) Wet the inside of a large test tube with Liquor Ammonia. Now drop into the tube some wood charcoal previously heated in a covered crucible. Cork the tube and after a few minutes remove the cork, and ascertain by smelling it whether there is any Ammonia left in the test tube.

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**14.—CARBON DIOXIDE.—Symbol, CO<sub>2</sub>. Mol. Wt., 44.**

PREPARATION.—Place some pieces of marble (Calcium Carbonate) in a flask with funnel and conducting tube, pour some water over it, and then a little Hydrochloric Acid; a rapid effervescence will begin, and the gas will be given off copiously. It may be collected over water, but as it is much heavier than air it is best collected by downward displacement. This is done by leading the conducting tube to the bottom of the gas jars, and allowing the heavy gas to collect in them and displace the air. As the gas extinguishes flame, the jars are ascertained to be full when a lighted taper placed in the mouth is at once extinguished. When this is the case cover the jar and substitute another; in this way collect four jars of the gas.

PROPERTIES.—(a) Place a lighted taper in the gas. What takes place?

(b) Devise an experiment for proving whether CO<sub>2</sub> is lighter or heavier than air.

(c) Allow the gas to bubble through lime water Ca(OH)<sub>2</sub>. What is formed? Continue to pass the gas through the liquid. What is the result? What is the reason?

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15.—CARBON MONOXIDE. Symbol, CO. Mol. Wt., 28.

PREPARATION.—Into a Florence flask put 8 or 10 grains of Oxalic Acid, and about 50 c.c. of Sulphuric Acid. Fit with a tight cork and tube and attach to a wash bottle containing a strong solution of Caustic Potash. From the wash bottle a delivery tube should pass to the pneumatic trough. Apply heat cautiously to the flask, regulating it so that the gas may come off in a steady stream. After the air has been expelled from the apparatus, collect three bottles of the gas, and allow these bottles to stand over water for some time before using them. Meanwhile substitute for the delivery tube one whose end has been drawn to a fine point. Apply a lighted match to the jet. Why is the wash bottle used? Write the equation.

PROPERTIES.—(a) Raise one of the bottles of gas from the water, and apply a lighted taper to its mouth.

(b) Try to pour the gas from one bottle to another, then test the result with a lighted paper.

(c) Purify thoroughly the gas in a third bottle, by shaking it up well with Caustic Potash or Caustic Soda solution, then test the gas with clear Lime-water. Compare the properties of the two Oxides of Carbon.

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16.—METHANE. Symbol, CH<sub>4</sub>. Mol. Wt., 16.

Take a hard glass test-tube or Florence flask, and fit with a cork and fine delivery tube. Place in the test-tube two grams of Acetate of Sodium NaC<sub>2</sub>H<sub>3</sub>O<sub>2</sub>, 8 grams of Sodium Hydroxide and 2 grams of finely powdered Quicklime, CaO, heat. After collecting a beaker or two of the gas, light the jet and observe the colour of the flame.

Fill a small soda water bottle with a mixture of one part of the gas and two parts of Oxygen. Ignite the mixture. Express the reaction by an equation.

Take a stoppered bottle and fill it with a mixture of equal volumes of Marsh Gas and Chlorine. Expose to sunlight for a day, then test the contents with blue litmus. Note any change in colour. Write the equations for the above reactions. What are the properties of Methane? How does it occur naturally?

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**17.—ETHYLENE (Ethene, Olefraut Gas). Symbol,  $C_2H_4$ . Mol. Wt., 28.**

Into a flask provided with a delivery tube and thistle tube pour 50 to 60 cc. of strong  $H_2SO_4$  and half the quantity of Alcohol. Heat gently. Collect the gas which comes off over water. Write the equation. Is the action of the  $H_2SO_4$  in this case comparable with its action in any previous experiment?

Will the gas burn? Try it.

Devise simple experiments to prove that the gas contains Hydrogen and Carbon.

Find out whether the gas is heavier or lighter than air.

Remove a jar of the gas, let it drain well, then turn it mouth upward and place over it another jar of the same size filled with Chlorine. After standing for some time, note whether the colour of the Chlorine changes. Observe closely what forms at the bottom of the lower jar—"Dutch liquid."

Ascertain whether the gas will explode when mixed with air or oxygen.

**18.—ACETYLENE. Symbol,  $C_2H_2$ . Mol. Wt., 30.**

In an evaporating dish place some pieces of Calcium Carbide. Wet these with water from a wash bottle. What takes place? Smell the gas which comes off. Will it burn?

Devise an experiment to find out whether it forms an explosive mixture with air. Write the equations. How is Calcium Carbide manufactured?

**19.—SULPHUR DIOXIDE. Symbol,  $SO_2$ . Mol. Wt., 64.**

Put 20 grams of copper turnings into a Florence flask, fitted up like a Hydrogen generating apparatus. Just cover the copper with strong Sulphuric Acid, and heat the flask very carefully. Collect the gas that escapes by downward displacement of the air. Compare its odour with that of burning sulphur. Collect three jars of the gas for further experiments. Keep them covered.

Pour some water into one of the jars and then shake. Is the gas soluble in water? Test the water with blue litmus solution. Taste it.

Hang a red rose or other high-coloured flower in the second jar. If any change takes place in the flower, remove it and place it in pure air.

Pour the third jar of Sulphur Dioxide into an "empty" jar with a burning candle at its bottom.

Write the equation. What are the properties of Sulphur Dioxide?

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**SULPHURETTED HYDROGEN.** Symbol,  $H_2S$ , Mol Wt. 34.

PREPARATION.—Take a Hydrogen generating apparatus, and place in it some powdered Sulphide of Iron. Cover the Sulphide with water, cork the apparatus tightly, and then add a few drops of Sulphuric Acid. Collect two or three bottles of the gas over warm water. Do not allow much of the gas to escape into the room. It is poisonous.

Write the equation.

Bubble the gas through a solution of blue litmus.

Devise an experiment to ascertain whether the gas is soluble in water.

Place in a test glass a solution of Copper Sulphate, in another a solution of Iron Sulphate, and in a third a solution of Sodium Chloride; to each add a few drops of Hydrochloric Acid, and then a little Sulphuretted Hydrogen water. Observe the black precipitate of Copper Sulphide in the first glass, and no precipitate in the other two glasses. To each of these add a little potash solution, and observe the black precipitate of Ferrous Sulphide in the one case, and the absence of a precipitate in the other.

How may metals be classified on the basis of the solubility of their sulphides? How would you separate copper from iron contained in a solution?

Attach a piece of glass tubing drawn to a fine point to the generating apparatus and try whether the gas will burn. What are the products of combustion.

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## SECTION II.

### EXAMINATION OF SUBSTANCES IN A DRY WAY.

#### A.—ASSAY IN A GLASS TUBE CLOSED AT ONE END.

##### I.—The Substance is Completely or Partially Volatilized.

1. Water is given off, which condenses in the upper part of the tube. This is examined with test paper. An alkaline reaction indicates Ammonia, an acid reaction the presence of a volatile acid such as  $\text{H}_2\text{SO}_4$ ,  $\text{HCl}$ ,  $\text{HNO}_3$ , or  $\text{HF}$ .

2. Gas or vapour is evolved.

(a) *Oxygen*, detected by means of a glowing splinter held in the mouth of the tube, indicates Peroxides, Nitrates, Chlorates, Bromates, or Iodates. Example,  $\text{KClO}_3$ .

(b) *Sulphur Dioxide*, detected by its odour and its acid reaction, indicates Sulphates or Sulphides. Example,  $\text{CuSO}_4$ .

(c) *Hydrogen Sulphide* denotes Sulphides containing water.

(d) *Nitrogen Tetroxide*, recognized by its colour and odour, indicates Nitrates or Nitrites. Ex.  $\text{Pb}(\text{NO}_3)_2$ .

(e) *Carbon Dioxide* is a decomposition product of Carbonates and some Oxalates. It will turn a drop of lime water held on the end of a glass rod milky.

(f) Ammonia indicates Ammonium Salts. Ex., Microcosmic Salt, or organic substances containing Nitrogen.

(g) *Hydrofluoric Acid* etches the glass tube.

3. A Sublimate is formed.

##### (a)—WHITE SUBLIMATES.

(a) Ammonium Salts. If the sublimate be removed from the tube and heated with a little  $\text{Na}(\text{OH})$  and a drop of water on platinum foil, Ammonia is evolved. Ex.  $\text{NH}_4\text{Cl}$ .

(b) *Chlorides of Mercury*.  $\text{HgCl}$  sublimes without previous fusion, whilst  $\text{HgCl}_2$  first fuses. The sublimate is yellow, hot, but white on cooling.





(c) *Antimony Trioxide* fuses to a yellow liquor, then sublimes to needle-shaped crystals.

(d) *Arsenic Trioxide* sublimes to octahedral crystals.

(b)—GREY OR BLACK SUBLIMATES.

(a) *Metallic Arsenic* and certain Arsenides.

(b) *Mercury, amalgams,* and some Mercury Salts.

(c) Some Cadmium alloys.

(c)—COLOURED SUBLIMATES.

(a) *Sulphur* and certain *Sulphides* which contain much Sulphur yield a deep yellow to brownish red sublimate when hot, sulphur yellow when cold. Ex., Pyrite.

(b) *Antimony Sulphide* when heated to a very high temperature yields a sublimate which is black while hot and reddish-brown when cold.

(c) *Arsenic Sulphides* yield sublimes dark reddish brown, hot, red to reddish-yellow, cold.

(d) *Cinnibar* yields a black sublimate which becomes red when rubbed. If heated strongly it volatilizes entirely.

II.—Substance Changes without Volatilization.

(1) Change of colour.

(a) White to yellow, white on cooling; Zinc Oxide.

(b) White to yellowish brown, dirty pale yellow, cold; Tin Oxide.

(c) White to brownish red, yellow cold; Lead Oxide (fusible).

(d) White to orange yellow or reddish brown, pale yellow cold; Bismuth Oxide (fusible).

(e) From red to black, red cold, Mercuric Oxide (volatile).

(f) From red to black, red cold; Ferric Oxide (non-volatile).

(2) *Fusion*: Alkaline salts.

(3) *Carbonization*: Organic substances.

(4) *Phosphorescence*: Alkaline earths, earths, zinc oxide, tin oxide, and many minerals.

(5) *Decrepitation*: Alkaline chlorides, galena and many minerals.

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## B.—ASSAY OF THE SUBSTANCE ON CHARCOAL.

(1) *Fusibility*.—Most of the alkaline salts and some of the salts of the alkaline earths are easily *fusible*. The following are *infusible* without flame colouration: Compounds of the earths and of the alkaline earths, silica and many silicates. The earths and alkaline earths emit white light when heated.

*Infusible* with change of colour: Zinc oxide, and tin oxide, which attain a temporary yellow colour.

The following metals are *easily fusible*:—Antimony, lead, cadmium, bismuth, zinc, and tin.

The following are with more *difficulty fusible*: copper, gold and silver. Iron, nickel and platinum are *infusible* in the blow pipe flame.

(2) *Decrepitation*.—Common salt and other haloid salts, substances containing mechanically included water; and many minerals.

(3) *Deflagration*.—Nitrates, chlorates, iodates, bromates.

(4) *Intumescence*.—Substances containing water, also borates and alum.

(5) *Odour*.—Sulphur Dioxide indicates a sulphide; odour of garlic indicates arsenic.

(6) Metal reduction and formation of incrustation.

(a)—REDUCED METAL WITHOUT INCRUSTATION.

Gold, silver and copper yield *glistening malleable beads*.

Platinum, iron, nickel, cobalt give a *grey infusible powder*, which in the case of the last three is magnetic.

(b)—REDUCED METAL WITH INCRUSTATION.

*Antimony* fuses easily and covers the charcoal with a white incrustation situated not far from the assay. If metallic antimony be fused and heated to redness it will burn, giving off white fumes. Drop a hot bead of antimony upon a sheet of paper and note the phenomenon.

*Bismuth* fuses easily in both flames. The incrustation is orange yellow hot and lemon yellow cold.

*Lead*, which fuses easily, incrusts the charcoal, in both flames, with oxide, which is lemon yellow hot, sulphur yellow cold.

*Tin* fuses very easily. In the oxidizing flame it is converted into the oxide, which is yellow, hot, white, cold and non-volatile in both flames. In the reducing flame the fused metal retains its metallic lustre.

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*Silver.* Silver Oxide is reduced to a brilliant metallic bead, which, if heated for a long time in a strongly oxidizing flame, gives a dark red incrustation. If the silver be mixed with lead or antimony, a yellow or white incrustation appears before the red one; if lead and antimony both are present at the same time, the incrustation has an intense rose colour.

(c)—INCRUSTATION WITHOUT METAL.

*Arsenic* volatilizes without previous fusion, giving a white incrustation which has a characteristic garlic odour. The incrustation disappears before both flames.

*Zinc* fuses easily and burns in the oxidizing flame with an intensely luminous, greenish white flame, giving off dense white fumes. The incrustation is yellow hot and white cold.

*Cadmium* fuses easily and burns in the oxidizing flame to brown Oxide. The flame is coloured dark yellow. The incrustation is reddish brown to orange yellow in thin layers, and is volatilized in both flames.

C.—EXAMINATION WITH BORAX.

Many metallic oxides, when heated with Borax to a high temperature, are dissolved by it, imparting a characteristic colour. A loop is made in the end of a platinum wire. This is heated in the blow pipe flame and dipped in powdered Borax, which is heated in the flame until a transparent glass bead appears in the loop. This is dipped into the material under examination, and the bead is heated in the oxidizing and reducing flames, when results in accordance with the following table should be obtained. *Be very careful to use the smallest possible amounts of substances on your borax bead in order to obtain the best results.*

IN THE OXIDIZING FLAME.		IN THE REDUCING FLAME.		INDICATION.
Hot.	Cold.	Hot.	Cold.	
Yellow to red.	Colourless to yellow.	Green.	Bottle green.	Iron oxide.
Yellow to red.	Grass green.	Green.	Emerald green.	Chromium oxide.
Violet	Reddish brown.	Grey c. bl. colourless.	Grey c. bl. colourless.	Nickel oxide.
Violet s. bl. black	Reddish violet s. b. black.	Colourless.	Colourless.	Manganese oxide.
Blue.	Blue.	Blue.	Blue.	Cobalt oxide.
Green.	Bluish green	Colourless.	Brown c. bl. red.	Copper oxide.

Contractions c. bl. continued blowing; s. b. saturated bead.

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## D.—FLAME COLOURATION.

When placed in a flame many elements impart a characteristic colour. To exhibit this dip a *clean* platinum wire into a salt of the metal, preferably the chloride. This is placed carefully in the colourless outer mantle of a Bunsen flame. The following are the most important characteristic colours.

## RED FLAMES.

Lithium, carmine red.  
 Strontium, scarlet red. } Barium salts obscure the reaction.  
 Calcium, yellowish red. }

## YELLOW FLAMES.

Sodium, orange yellow.

## GREEN FLAMES.

Copper Oxide, emerald green, after moistening with HCl, blue.  
 Phosphoric acid, bluish green. } In their salts after moisten-  
 Boric acid, yellowish green. } ing with  $H_2SO_4$ .  
 Barium salts, yellowish green.  
 Antimony, faint green.  
 Nitric acid, bronze green, quickly disappearing.

## BLUE FLAMES.

Copper Chloride, azure blue, afterwards green.  
 Arsenic, bluish.  
 Lead, blue.

## VIOLET FLAMES.

Potassium, violet red. Sodium salts obscure the reaction. If, however, the flame be looked at through blue glass the yellow light rays of sodium will be cut off and the violet rays only will pass through. In this way Potassium when obscured by Sodium may be detected.

## E.—EXAMINATION WITH COBALT NITRATE.

Certain substances when treated with cobalt nitrate exhibit certain characteristic colours. The substance to be examined is heated on charcoal, then moistened thoroughly with Cobalt Nitrate solution, having been previously pulverized if necessary, and then heated. The colours produced are :

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Green, Zinc Oxide, yellowish green.  
" Tin Oxide, bluish green.  
" Antimony Oxide, dirty green.  
Flesh-red, Magnesia, pale flesh red.

F.—REACTIONS WITH HYDRIODIC ACID.

A hole is made at one end of a Plaster Paris tablet. In this some of the substance to be examined is placed and moistened with Hydrionic Acid. When heated in the oxidizing flame the following incrustations appear :

Antimony, orange red.

Arsenic, buff.

Lead, chrome yellow.

Tin, brownish yellow.

Silver, *Hot*, light yellow; *Cold*, faint greyish yellow (close to the assay).

Mercury, yellow and scarlet; the yellow becoming scarlet on standing or on being rubbed.

Bismuth, chocolate brown.

Cobalt, greenish brown.

Copper, white.

Zinc, white.

Cadmium, white.

NOTE —In the case of *White* incrustations the tablet should first be smoked.

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## SECTION III.

### QUALITATIVE ANALYSIS IN THE WET WAY.

#### Reactions of the Metals of the Silver Group.†

Metals whose chlorides are insoluble in water, and which are precipitated on addition of the group reagent, HCl:

#### Silver, Mercury, Lead.

##### SILVER. AG. 108. SOLUTION FOR REACTIONS, $\text{AgNO}_3$ .

1. HCl produces a white curdy precipitate of HCl, insoluble in hot water and in  $\text{HNO}_3$ , but readily soluble in  $(\text{NH}_4)\text{HO}$ .
2.  $\text{H}_2\text{S}$ , or  $(\text{NH}_4)_2\text{S}$ , produces a black precipitate of  $\text{Ag}_2\text{S}$ , soluble in boiling  $\text{HNO}_3$ , with separation of sulphur.
3. NaHO produces a light brown precipitate of  $\text{Ag}_2\text{O}$ , insoluble in excess of NaHO, but soluble in  $(\text{NH}_4)\text{HO}$ .
- \*4.  $\text{K}_2\text{CrO}_4$  produces a dark red precipitate of  $\text{Ag}_2\text{CrO}_4$ , soluble in hot  $\text{HNO}_3$ ; this solution deposits on cooling an acid chromate in needle-shaped crystals.
5. KI produces a pale yellow precipitate of AgI insoluble in  $\text{HNO}_3$ .
6. Heated on charcoal with  $\text{Na}_2\text{CO}_3$ , in the reducing flame of the blow-pipe, yields bright, malleable metallic beads, soluble in  $\text{HNO}_3$ .

Write the equations for the above reactions.

##### MERCURY. Hg. 200. MERCUROUS SALTS. SOLUTION FOR REACTIONS, $\text{Hg}(\text{NO}_3)_2$ .

1. HCl produces a white precipitate of HgCl (calomel), insoluble in cold  $\text{HNO}_3$ , and blackened by  $(\text{NH}_4)\text{HO}$ , from a formation of  $\text{Hg}_2\text{Cl}(\text{NH}_2)$ .

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† The best confirmatory tests are indicated in the following pages by an asterisk.

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2.  $\text{H}_2\text{S}$  or  $(\text{NH}_4)_2\text{S}$  produces a black precipitate of  $\text{Hg}_2\text{S}$ , not dissolved by boiling  $\text{HNO}_3$ .

3.  $\text{NaHO}$  precipitates black  $\text{Hg}_2\text{O}$ , insoluble in excess of  $\text{NaHO}$  or  $(\text{NH}_4)\text{HO}$ ; decomposes readily into  $\text{HgO}$  and  $\text{Hg}$ .

How would you separate Mercury from Lead in a solution ?

\*4.  $\text{SnCl}_2$  precipitates grey  $\text{Hg}$ . If the fluid be poured off, and the residue boiled with  $\text{HCl}$ , distinct globules are obtained.

5.  $\text{KI}$  precipitates dark-green  $\text{HgI}$ .

6. A drop of a metal or only slightly acid solution of a mercurous salt placed on a bright copper coin will deposit mercury, and the stain will become bright by rubbing.

7. Heated in small tube with  $\text{NaHCO}_3$ , yields grey deposit of  $\text{Hg}$ .  $\text{Hg}$  is volatile, and condenses on the cooler parts of the tube; soluble in  $\text{HNO}_3$ .

Write the equations.

#### LEAD, $\text{Pb}$ , 207. SOLUTION FOR REACTION, $\text{Pb}(\text{NO}_3)_2$

1.  $\text{HCl}$  precipitates (incompletely) white  $\text{PbCl}_2$ , soluble in boiling  $\text{H}_2\text{O}$ , or in large quantity of cold  $\text{H}_2\text{O}$ ; converted into a basic salt on adding  $(\text{NH}_4)\text{HO}$ , without change of appearance. If  $\text{PbCl}_2$  be dissolved in boiling  $\text{H}_2\text{O}$ , it will crystallize from this solution on cooling.

Supposing you had silver, mercury and lead in solution, how could you separate the lead from the other two ?

\*2.  $\text{H}_2\text{SO}_4$  precipitates heavy white  $\text{PbSO}_4$ , soluble in  $\text{NaHO}$  or ammonium tartrate. This precipitate in dilute solutions only appears on standing. If there is no immediate precipitation, concentrate the solution by evaporation.  $\text{PbSO}_4$  is soluble in boiling  $\text{HCl}$ , and the solution, on cooling, deposits needle-shaped crystals of  $\text{PbCl}_2$ .

$\text{H}_2\text{S}$  produces a black precipitate of  $\text{PbS}$  insoluble in  $\text{HCl}$ .

3.  $\text{K}_2\text{CrO}_4$  produces a bright yellow precipitate of  $\text{PbCrO}_4$ , readily soluble in  $\text{NaHO}$ , but with difficulty in  $\text{HNO}_3$ .

\*4.  $\text{KI}$  produces a bright yellow precipitate of  $\text{PbI}_2$ , soluble in boiling water; the solution on cooling deposits the salt in brilliant golden hexagonal scales.

5. Heated on charcoal with  $\text{NaHCO}_3$ , yields malleable beads, and at the same time a yellow incrustation of  $\text{PbO}$  on the charcoal.

Write the equations. Why are  $\text{Ag}$ ,  $\text{Pb}$  and  $\text{Hg}$  placed in one group ?

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TABLE A—SILVER GROUP (1).

## Separation of Silver, Mercury and Lead.

Add HCl and filter from the precipitated chlorides.

PRECIPITATE.

FILTRATE.

AgCl, HgCl, PbCl<sub>2</sub>.

Groups II, III, IV, &amp; V.

Wash precipitate twice with cold water, and add washings to filtrate, then twice with hot water, and test part of this for lead with dilute H<sub>2</sub>SO<sub>4</sub>. White precipitate indicates Lead. Boil the remaining part down to obtain the needle-shaped crystals of PbCl<sub>2</sub>. If lead be found, wash the precipitate free from it with hot water, and treat the residue repeatedly with warm (NH<sub>4</sub>) HO; filter.

RESIDUE.

FILTRATE

Hg.

Ag.

If the residue is black, this indicates Mercury. Dissolve in HCl + HNO<sub>3</sub> and test with Sn Cl<sub>2</sub>. Confirm by reduction test.

Add HNO<sub>3</sub>. A white precipitate indicates the presence of Silver. Confirm with blow-pipe.

## GROUP II.—(COPPER GROUP.)

Group reagent, H<sub>2</sub>S in presence of HCl.

Mercury, Lead, Bismuth, Copper, Cadmium, Arsenic, Antimony, and Tin.

The sulphides of the metals of this group are insoluble in HCl, and are therefore precipitated by H<sub>2</sub>S in an aqueous solution containing HCl. The three last metals, arsenic, antimony, and tin, form a sub-group, as their sulphides are soluble in (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>, whilst the sulphides of the remaining metals are insoluble in that reagent.

SUB-GROUP A.—Sulphides of the above metals insoluble in (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>, viz.: Mercury, Lead, Copper, Bismuth, and Cadmium.

MERCURY, Hg 200 (MERCURIC SALTS). SOLUTION FOR REACTIONS, HgCl<sub>2</sub>.

1. H<sub>2</sub>S produces, when added by degrees, first a white precipitate, which changes to orange, then to brownish-red, and finally to a black precipitate of HgS. These successive changes of colour on the addition are exceedingly characteristic. This precipitate is insoluble in HCl and in HNO<sub>3</sub>, even on boiling; it is soluble in KHS and aqua regia.

How is HgS separated from other sulphides of this group?

2. KHO produces a yellow precipitate of HgO insoluble in excess, except when added to very acid solutions.

3. (NH<sub>4</sub>)HO produces a white precipitate of HgCl(NH<sub>2</sub>) (white precipitate.)

\*4. SnCl<sub>2</sub> when added in small quantities precipitates a white Hg<sub>2</sub>Cl<sub>2</sub>; when added in excess, gray metallic Hg, which may be united into a globule by boiling with HCl.

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5. KI precipitates bright-red  $\text{HgI}_2$ , soluble in excess of either KI or  $\text{HgCl}_2$ .

6.  $\text{K}_2\text{CrO}_4$  precipitates an orange basic chromate easily soluble in  $\text{HNO}_3$ .

Write the equations. How would you distinguish between mercurous and mercuric salts?

LEAD. Pb 207. SOLUTION FOR REACTIONS  $\text{Pb}(\text{NO}_3)_2$ .

1.  $\text{H}_2\text{S}$  produces a black precipitate of  $\text{PbS}$ , even in solutions of  $\text{PbCl}_2$ , so that a weak solution of a lead salt which has not been precipitated with  $\text{HCl}$  will be precipitated with  $\text{H}_2\text{S}$ . Hence lead occurs both in the silver and copper groups.

2. Reaction, 2, 3, 4, and 5, for lead, in Group I., are also applicable in this group.

How may lead be separated from the rest of the group?

BISMUTH. Bi. 210. SOLUTION FOR REACTIONS.  $\text{BiCl}_3$ .

1.  $\text{H}_2\text{S}$  produces a black precipitate of  $\text{Bi}_2\text{S}_3$ , insoluble in  $\text{KHS}$  and  $\text{KHO}$ , but soluble in  $\text{HNO}_3$ .

2.  $\text{KHO}$  or  $(\text{NH}_4)\text{HO}$  produces a white precipitate, which on boiling becomes yellow ( $\text{Bi}_2\text{O}_3$ ); the precipitate is insoluble in excess of either reagent.

\*3.  $\text{H}_2\text{O}$ , when added in considerable quantity to normal salts of Bismuth, produces an immediate white precipitate of basic salt of Bismuth.

Bismuth Trichloride is most easily precipitated by  $\text{H}_2\text{O}$ . If another salt of this metal is being examined, it is best to precipitate the oxide first by ammonia; dissolve it in as little  $\text{HCl}$  as possible, and evaporate it almost to dryness. On adding water to this solution, a precipitate of  $\text{BiOCl}$  at once forms, which is insoluble in Tartaric Acid (compare corresponding reaction with Antimony). Solutions of Bismuth Salts containing much free acid do not give this reaction with  $\text{H}_2\text{C}$  until the excess of acid has been expelled by evaporation.

4.  $\text{K}_2\text{CrO}_4$  produces a yellow precipitate of  $\text{Bi}_2(\text{CrO}_4)_3$ , soluble in  $\text{HNO}_3$ , and insoluble in  $\text{NaHO}$ .

5.  $\text{Zn}$  or  $\text{Fe}$  precipitates spongy  $\text{Bi}$ .

\*6. Heated on charcoal with  $\text{Na}_2\text{CO}_3$ , in the reducing blowpipe

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flame, compounds of Bi yield brittle metallic globules ; also a yellow incrustation of  $\text{Bi}_2\text{O}_3$  on the Charcoal. Bi is soluble in  $\text{HNO}_3$  or aqua regia.

Write the equations.

COPPER, Cu, 63.5. SOLUTION FOR REACTIONS,  $\text{CuSO}_4$ .

1.  $\text{H}_2\text{S}$  precipitates black,  $\text{CuS}$ , soluble in  $\text{HNO}_3$  ; insoluble in  $\text{KHS}$ , and only slightly soluble in  $(\text{NH}_4)_2\text{S}_2$ .  $\text{CuS}$  is also soluble in  $\text{KCN}$ , but insoluble in hot dilute  $\text{H}_2\text{SO}_4$ .

2.  $\text{KHO}$  precipitates a pale-blue  $\text{Cu}(\text{HO})_2$ , insoluble in excess. If  $\text{KHO}$  be added in excess and the mixture boiled, the precipitate becomes black.

3.  $(\text{NH}_4)\text{HO}$  precipitates, when added in small quantities, greenish blue basic salt, soluble in excess of  $(\text{NH}_4)\text{HO}$ , forming a dark blue solution which consists of double basic salt of copper and ammonium.

4.  $\text{K}_4\text{Fe}(\text{CN})_6$  precipitates brown  $\text{Cu}_2\text{Fe}(\text{CN})_6$ , insoluble in dilute acids, but decomposed by  $\text{KHO}$ .

5.  $\text{Fe}$  precipitates  $\text{Cu}$  in the metallic state, especially in the presence of a little free acid.

6.  $\text{Zn}$  also precipitates copper solutions.

7.  $\text{K}_2\text{CrO}_4$ , precipitates a brownish-red basic chromate, soluble in  $\text{HNO}_3$  and in  $(\text{NH}_4)\text{HO}$ .

8. Compounds of  $\text{Cu}$ , when heated in Bunsen flame, impart a green colour, especially after addition of  $\text{AgCl}$ .

9. Heated on charcoal with  $\text{NaHCO}_3$  in reducing flame, yields brittle metallic globules of bright-red colour, soluble in  $\text{HNO}_3$  or concentrated  $\text{H}_2\text{SO}_4$ .

Write the reactions.

CADMIUM, Cd, 112. SOLUTION FOR REACTIONS,  $\text{Cd}(\text{NO}_3)_2$

1.  $\text{H}_2\text{S}$  precipitates yellow  $\text{CdS}$ , soluble in  $\text{HNO}_3$ , insoluble in  $\text{KHS}$ ,  $\text{KCN}$ , and  $(\text{NH}_4)_2\text{S}$ .  $\text{CdS}$  is dissolved by hot dilute  $\text{H}_2\text{SO}_4$ . Compare with copper reaction.

2.  $\text{KHO}$  precipitates  $\text{Cd}(\text{HO})_2$ , insoluble in excess of reagent.

3.  $(\text{NH}_4)\text{HO}$  precipitates  $\text{Cd}(\text{HO})_2$  ; soluble in excess of reagent.

4.  $\text{Zn}$  precipitates  $\text{Cd}$  in brilliant scales.

5. Heated on charcoal with  $\text{NaHCO}_3$  in reducing flame, yields a brown incrustation of  $\text{CdO}$ .  $\text{Cd}$  dissolves readily in  $\text{HNO}_3$ .

Write equations. How may copper and cadmium be separated ?

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TABLE B.—COPPER GROUP (II).

## Separation of Mercury, Lead, Bismuth, Copper, and Cadmium (Sub-Group A.)

To the filtrate from the Silver Group add an equal bulk of HCl, boil down nearly to dryness, (why?) dilute with H<sub>2</sub>O, and pass H<sub>2</sub>S through the hot solution. Filter.

RESIDUE.		FILTRATE.
HgS, PbS, Bi <sub>2</sub> S <sub>3</sub> , CuS, CdS, SnS, SnS <sub>2</sub> , Sb <sub>2</sub> S <sub>3</sub> , and As <sub>2</sub> S <sub>3</sub> .	}	Groups III, IV., and V.

Wash with hot H<sub>2</sub>O containing H<sub>2</sub>S, until free from Cl; [digest residue\* with (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub> for about fifteen minutes. Filter.

RESIDUE		FILTRATE.
HgS, PbS, Bi <sub>2</sub> S <sub>3</sub> , CuS, and CdS.	}	Sub-Group B.
		(Sn Sb, and As.)
		(See Table E.)

\*In the absence of the sub-group omit that which is in parenthesis, and treat with HNO<sub>3</sub>, as below.

Wash with hot H<sub>2</sub>O till no longer alkaline; add a small quantity of boiling HNO<sub>3</sub>, pouring it on several times. Filter.

RESIDUE.		FILTRATE.
HgS.	}	Pb, Bi, Cu, Cd.
Dissolve in aqua regia, boil down to expel acid, and test with SnCl <sub>2</sub> . White precipitate changing to grey indicates Mercury. Confirm by reduction test.		Add H <sup>2</sup> SO <sub>4</sub> , and boil down. White precipitate indicates Lead. Filter from PbSO <sub>4</sub> , and add (NH <sub>4</sub> )HO to filtrate. Filter.

RESIDUE.		FILTRATE.
Bi.	}	Cu, Cd.
A white precipitate. Dissolve in HCl, evaporate to a small bulk, and add to H <sub>2</sub> O. A white precipitate indicates Bismuth.		A blue solution indicates Copper. Adopt Method I. or II.

## METHOD I.

Add to the blue solution KCN till colourless; pass a few bubbles of H<sub>2</sub>S through the solution. Yellow precipitate indicates Cadmium. (Excess of H<sub>2</sub>S must be avoided, because traces of mercury may be present.)

## METHOD II.

Add HCl till acid; pass H<sub>2</sub>S through; filter; wash rapidly and thoroughly, and treat with hot dilute H<sub>2</sub>SO<sub>4</sub>. Black residue indicates Copper. To filtrate, add H<sub>2</sub>S. Yellow precipitate indicates Cadmium.

NOTE.—The student should be able to trace by means of equations the separation of all the elements of this and other groups. He should, also, practice writing out on paper the separation of mixtures of two or more metals and amalgams.

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SUB-GROUP B. SULPHIDES SOLUBLE IN  $(\text{NH}_4)_2\text{S}$ , VIZ.: THOSE OF  
TIN, ANTIMONY AND ARSENIC.

TIN. Sn 118 (STANNOUS SALTS) SOLUTION FOR REACTIONS  $\text{SnCl}_2$ .

1.  $\text{H}_2\text{S}$  precipitates dark brown  $\text{SnS}$ , soluble in yellow  $(\text{NH}_4)_2\text{S}_2$  (nearly insoluble in colourless  $(\text{NH}_4)_2\text{S}$ ; re-precipitated as yellow  $\text{SnS}_2$  by  $\text{HCl}$ .
2.  $\text{KHO}$  precipitates  $\text{Sn}(\text{HO})_2$ , soluble in excess of reagent.
3.  $(\text{NH}_4)\text{HO}$  precipitates  $\text{Sn}(\text{HO})_2$ , not soluble in excess.
- \*4.  $\text{HgCl}_2$  precipitates at first white  $\text{HgCl}$ ; on boiling with excess of reagent, grey  $\text{Hg}$ .
5.  $\text{AuCl}_3$ , on addition of a little  $\text{HNO}_3$ , precipitates purple (royal purple of Cassius).
6.  $\text{Zn}$  precipitates metallic tin.
7. Mixed with  $\text{NaHCO}_3 + \text{KCN}$ , and heated on charcoal in reducing flame, yields small globules of  $\text{Sn}$  and a white incrustation of  $\text{SnO}_2$ .

STANNIC SALTS, Sn. SOLUTION FOR REACTIONS,  $\text{SnCl}_4$ .

Stannous Salts are converted into Stannic by oxidizing agents,  $\text{HNO}_3$ ,  $\text{Cl}$ ,  $\text{FeCl}_3$ , etc.

1.  $\text{H}_2\text{S}$  precipitates yellow  $\text{SnS}_2$ , soluble in  $(\text{NH}_4)_2\text{S}$ , in  $\text{KHO}$ , and in boiling concentrated  $\text{HCl}$ . It is with difficulty soluble in  $(\text{NH}_4)\text{HO}$ , and insoluble in  $(\text{NH}_4)_2\text{CO}_3$ .
2.  $\text{KHO}$  or  $(\text{NH}_4)\text{HO}$  precipitates white  $\text{SnO}(\text{HO})_2$ , soluble in an excess of precipitant.
3.  $\text{Zn}$  produces same reaction as with Stannous Salts.
4. The blow-pipe reaction for Stannic is the same as for Stannous.

How are Stannous distinguished from Stannic Salts? Write the equations.

ANTIMONY, Sb, 122.3. SOLUTION FOR REACTIONS,  $\text{SbCl}_3$ .

1.  $\text{H}_2\text{S}$  precipitates orange  $\text{Sb}_2\text{S}_3$ , soluble in  $(\text{NH}_4)_2\text{S}$ ,  $\text{NaHO}$ , and in hot concentrated  $\text{HCl}$ ; insoluble in  $(\text{NH}_4)_2\text{CO}_3$ .
2.  $\text{KHO}$  precipitates  $\text{Sb}_2\text{O}_3$ , soluble in excess of reagent.
3.  $(\text{NH}_4)\text{HO}$  precipitates  $\text{Sb}_2\text{O}_3$ , insoluble in excess.

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4.  $\text{H}_2\text{O}$  produces in solutions of  $\text{SbCl}_3$  a white precipitate of  $\text{SbOCl}$ , soluble in tartaric acid.

5.  $\text{Zn}$  in presence of  $\text{HCl}$  and platinum precipitates  $\text{Sb}$  as a black powder, which adheres to the platinum. The black stain on the platinum is not removed by  $\text{HCl}$ , but is immediately dissolved by warm  $\text{HNO}_3$ .

6. (Marsh's Test.) If a solution of  $\text{Sb}$  be placed in a flask in which hydrogen is being generated,  $\text{SbH}_3$  is given off as a gas, which is decomposed by heat,  $\text{Sb}$  being deposited. This is best done by holding in the  $\text{SbH}_3$  flame a piece of cold porcelain; a dull-black stain of metallic  $\text{Sb}$  will be deposited on it. Add to the stain on the porcelain a drop of  $\text{NaClO}$ ; the stain will remain undissolved.

*Be very careful that all the air is expelled from the apparatus before lighting.*

7. Heated with  $\text{NaHCO}_3$  on charcoal in the reducing blow-pipe flame, yields brittle globules of the metal and a white incrustation of  $\text{Sb}_2\text{O}_3$  on the charcoal.

#### ARSENIC. $\text{As}$ , 75.2. SOLUTION FOR REACTION. $\text{Na}_3\text{AsO}_4$ .

1.  $\text{H}_2\text{S}$  produces in acid solutions a yellow precipitate of  $\text{As}_2\text{S}_3$ , soluble in alkaline sulphides, in  $\text{KHO}$ , in  $\text{HNO}_3$ , and in  $(\text{NH}_4)_2\text{CO}_3$ , but nearly insoluble in boiling concentrated  $\text{HCl}$ . (Compare reactions for  $\text{Sb}$ .)

2.  $\text{AgNO}_3$  produces in *neutral* solutions of the arsenites a pale yellow precipitate of  $\text{Ag}_3\text{AsO}_3$ . This is best obtained by adding  $\text{AgNO}_3$  to an aqueous solution of  $\text{As}_2\text{O}_3$ , and then drop by drop a *very dilute solution* of  $(\text{NH}_4)\text{HO}$  prepared by adding one or two drops of ordinary  $(\text{NH}_4)\text{HO}$  to a test-tube full of  $\text{H}_2\text{O}$ . The precipitate is readily soluble in excess of  $(\text{NH}_4)\text{HO}$ , hence the necessity for using a very dilute solution of that reagent.

3.  $\text{CuSO}_4$  added under the same conditions as the  $\text{AgNO}_3$ , produces a pale green precipitate of  $\text{CuHASO}_3$  (Scheele's green), soluble in  $(\text{NH}_4)\text{HO}$ .

4. Acetic acid, added to solutions of  $\text{As}_2\text{O}_3$  and then  $\text{KHO}$  in slight excess, yields (after evaporation to dryness), on ignition in a small tube, oxide of cacodyl  $2(\text{As}(\text{CH}_3)_2)\text{O}$ , readily recognized by its powerful and characteristic odour. If  $\text{SnCl}_2$  be added to the contents of the tube after ignition, the equally characteristic smell of cacodyl chloride,  $\text{As}(\text{CH}_3)_2\text{Cl}$ , is observed. These experiments

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(and also Marsh's test (5) ) must be done with an *exceedingly small* quantity of substance, owing to the poisonous properties of the products.

\*5. Proceed exactly as in Marsh's test for Sb, substituting a solution of As for one of Sb, and observe the bluish flame with which the mixture of H and  $\text{AsH}_3$  burns, and also the production of white fumes of  $\text{As}_2\text{O}_3$ . Obtain, as in the case of Sb, stains on porcelain lids. Compare these with the antimony stains. Observe also the distinction in colour of the stains: dark brown or almost black in the case of Sb, and, when seen in thin films, pale brown and lustrous in the case of As.

Add to one of the stains on porcelain a drop of  $\text{NaClO}$ : it will be rapidly dissolved.

Make a full statement of the chemical distinctions between arsenic and antimony.

6. Reinsch's Test: Add to the solution of arsenic, HCl and a few strips of bright copper wire or foil: As is deposited on the copper, which may be removed from the solution, dried by filter paper, and heated in a dry test-tube to obtain the octahedral crystals of  $\text{As}_2\text{O}_3$ .

7. Place the dry arsenic compound in a bulb tube along with a mixture of equal parts of *dry*  $\text{Na}_2\text{CO}_3$  and KCN, and heat the bulb. A mirror of As will be formed on the inside of the tube.

TABLE C.—(SUB GROUP B).

Solution in  $(\text{NH}_4)_2\text{S}_3$  contains sulphides of As, Sb, and Sn. Add HCl until acid (Why?). The metals are re-precipitated as sulphides. Filter, wash precipitate with hot water till free from HCl; digest precipitate with one or two pieces of solid  $(\text{NH}_4)_2\text{CO}_3$  and  $\text{H}_2\text{O}$ . Filter.

RESIDUE.	FILTRATE.
$\text{SnS}_2, \text{Sb}_2\text{S}_3.$	As.
<p>Wash and dissolve in strong boiling HCl, dilute with water, filter, and divide the filtrate into two parts. In one place a piece of platinum foil and a fragment of zinc touching it. Sb forms a black stain on the platinum. Dissolve by warming with a few drops of <math>\text{HNO}_3</math>, dilute with water and add <math>\text{H}_2\text{S}</math>. An orange precipitate indicates <b>Antimony</b>. Boil the other portion of the filtrate for at least five minutes with some metallic copper. Pour off the liquid and add <math>\text{HgCl}_2</math>. White or grey precipitate indicates <b>Tin</b>.</p>	<p>Add HCl until acid; wash precipitated sulphide, and dissolve in HCl and a little <math>\text{KClO}_3</math>, boil down to a small bulk, and apply Marsh's test. Metallic mirror, yielding octahedral crystals on heating, indicates <b>Arsenic</b>. Dissolve in <math>\text{H}_2\text{O}</math>, and confirm by adding <math>\text{AgNO}_3</math> and dilute <math>(\text{NH}_4)\text{HO}</math>, to obtain yellow precipitate of <math>\text{Ag}_3\text{AsO}_4</math>.</p>

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## GROUP III.

## Reactions of the Metals of the Iron Group.

Metals whose sulphides and hydrated oxides are insoluble in water, and are precipitated on addition of the group reagent  $(\text{NH}_4)_2\text{S}$  in presence of  $(\text{NH}_4)\text{HO}$  and  $\text{NH}_4\text{Cl}$ .

## Iron, Nickel, Cobalt, Zinc, Aluminium, Manganese, and Chromium.

IRON. Fe, 56. FERROUS SALTS. SOLUTION FOR REACTIONS  $\text{FeCl}_2$ .

1.  $(\text{NH}_4)_2\text{S}$  produces a black precipitate of  $\text{FeS}$ , insoluble in alkalis, but soluble in  $\text{HCl}$ . In dilute solutions of ferrous salts  $(\text{NH}_4)_2\text{S}$  produces at first a green colour; on standing, however,  $\text{FeS}$  separates as a black precipitate.

2.  $\text{KHO}$  or  $(\text{NH}_4)\text{HO}$  produces a white precipitate of ferrous hydrate  $\text{Fe}(\text{HO})_2$ , which rapidly acquires a dirty green, and ultimately a reddish-brown colour, owing to absorption of oxygen and conversion into ferric hydrate  $\text{Fe}(\text{HO})_3$ .

3.  $(\text{NH}_4)_2\text{CO}_3$  or  $\text{Na}_2\text{CO}_3$  precipitates white  $\text{FeCO}_3$ , which rapidly darkens in colour.

4.  $\text{K}_4\text{Fe}(\text{CN})_6$  precipitates white  $\text{K}_2\text{Fe}_2(\text{CN})_6$ , which rapidly becomes blue by oxidation on  $\text{Fe}_5(\text{CN})_{12}$  (Prussian blue).

5.  $\text{K}_3\text{Fe}(\text{CN})_6$  precipitates "Turnbull's blue,"  $\text{Fe}_3\text{Fe}_2(\text{CN})_{12}$ .

6.  $\text{KCNS}$  produces no colouration.

7.  $\text{BaCO}_3$  produces no precipitate in cold solution of ferrous salts.

8. Ferrous compounds are converted into ferric by oxidizing agents, such as  $\text{HNO}_3$ ,  $\text{KClO}_3$ ,  $\text{HCl}$ ,  $\text{Cl}$ .

9. Fused with borax in the oxidizing flame, yellowish-red beads are produced; in the reducing flame the beads become green.

FERRIC SALTS, Fe. SOLUTION,  $\text{FeCl}_3$ .

1.  $\text{H}_2\text{S}$  in acid solutions produces a precipitate of sulphur, and the salt is reduced to protosalt:—



2.  $(\text{NH}_4)_2\text{S}$  precipitates black  $\text{FeS}$  and  $\text{S}$ , soluble in  $\text{HCl}$  and  $\text{HNO}_3$ .

3.  $(\text{NH}_4)\text{HO}$  or  $\text{KHO}$  precipitates reddish brown  $\text{Fe}(\text{HO})_3$ , insoluble in excess of reagent.

4.  $\text{K}_4\text{Fe}(\text{CN})_6$  precipitates "Prussian blue,"  $\text{Fe}_5(\text{CN})_{12}$ , insoluble in  $\text{HCl}$ , soluble in  $\text{C}_2\text{H}_2\text{O}_4$ .

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5.  $K_3Fe(CN)_6$  produces a reddish-brown colour.

6. KCNS produces even in dilute solutions a blood-red colouration, due to the formation of a soluble iron sulphocyanide. HCl does not destroy the colouration, but it is destroyed by  $C_2H_5O_2Na$ ,  $HgCl_2$ ,  $H_3PO_4$ , and by  $C_4H_6O_6$ .

7.  $BaCO_3$  precipitates ferric solutions completely as  $Fe(OH)_3$  mixed with basic salt.

8. The blow-pipe reactions are the same as for ferrous compounds.

How do you distinguish between ferrous and ferric salts?

#### NICKEL. Ni, 58.7. SOLUTION, $Ni(NO_3)_2$ .

1.  $(NH_4)_2S$  produces a black precipitate of  $NiS$ , slightly soluble in excess, forming a brown solution from which  $NiS$  is precipitated on boiling. The precipitate is very difficultly soluble in HCl, but dissolves in  $HNO_3$  and in aqua regia.

2. NaHO or KHO produces a light green precipitate of  $Ni(OH)_2$  insoluble in excess of the reagent, and unalterable in air.

3.  $(NH_4)HO$  produces also a precipitate of  $Ni(OH)_2$ , readily soluble in excess, yielding a blue fluid, which is reprecipitated by KHO or NaHO. Acid solutions, or those containing Salts of Ammonia, yield no precipitate with  $(NH_4)HO$ .

4. KCN precipitates yellowish-green  $Ni(CN)_2$ , soluble in excess and reprecipitated by HCl or  $H_2SO_4$ , and if boiled with a strong solution of NaClO, yields a black precipitate of  $Ni(OH)_3$ .

5.  $KNO_3$  in presence of  $C_2H_4O_2$  produces no precipitate.

6. Fused with borax compounds of Ni, yields reddish-yellow beads when hot in oxidizing flame. In reducing flame the bead becomes grey.

Characteristic reactions, 2, 3.

#### COBALT, Co, 58.7. SOLUTION, $Co(NO_3)_2$

1.  $(NH_4)_2S$  precipitates black  $CoS$ , insoluble in excess of reagent and in HCl, soluble in aqua regia.

2. KHO or NaHO precipitates blue basic salts, which turn green on exposure to air by oxidation.

3.  $(NH_4)HO$  precipitates the same as above, soluble in excess, yielding a reddish-brown fluid; reprecipitated by NaHO or KHO. Acid solutions, or those containing Salts of Ammonia, are not precipitated.

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4. KCN precipitates light-brown  $\text{Co}(\text{CN})_2$ , soluble in excess of the reagent by formation of  $2\text{KCN}$ ,  $\text{Co}(\text{CN})_2$ . This solution is reprecipitated by addition of  $\text{HCl}$  or  $\text{H}_2\text{SO}_4$ . If to the solution in excess of KCN a few drops of  $\text{HCl}$  be added, and the solution boiled for some time,  $\text{K}_3\text{Co}(\text{CN})_6$ , Potassium Cobaltic Cyanide, is formed which is not reprecipitated by  $\text{HCl}$  or  $\text{H}_2\text{SO}_4$ , nor by  $\text{NaClO}$ .

5.  $\text{KNO}_2$  added to Cobalt solutions with addition of Acetic Acid, precipitate, on standing, a yellow crystalline double salt.

6. Compounds of cobalt fused with borax in either blow pipe flame yield deep-blue beads.

#### ZINC, Zn, 65.2. SOLUTION $\text{ZnSO}_4$ .

1.  $(\text{NH}_4)_2\text{S}$  produces a white precipitate of  $\text{ZnS}$ , insoluble in excess of the reagent and in  $\text{KHO}$ , but soluble in the mineral acids.

2.  $\text{KHO}$  or  $\text{NaHO}$  produces a white precipitate of  $\text{Zn}(\text{HO})_2$ , soluble in excess of either reagent and in  $(\text{NH}_4)\text{HO}$ . This solution is reprecipitated by diluting and boiling, but is not precipitated by addition of  $\text{NH}_4\text{Cl}$ .

3.  $\text{Na}_2\text{CO}_3$  produces a white precipitate of basic carbonate, insoluble in excess of the reagent.

4.  $(\text{NH}_4)_2\text{CO}_3$  also precipitates the basic carbonate, but it is soluble in excess of the reagent.

5. Heated on charcoal with  $\text{Na}_2\text{CO}_3$  in the reducing blow-pipe flame, a yellow incrustation of  $\text{ZnO}$  is obtained, which becomes white when cold.

\*6. Heated on charcoal by the blow-pipe flame after moistening with  $\text{Co}(\text{NO}_3)_2$  solution, an infusible green mass is obtained.

#### ALUMINIUM, Al, 27.3. SOLUTION, $\text{Al}_2(\text{SO}_4)_3$ .

1.  $(\text{NH}_4)_2\text{S}$  produces a white flocculent precipitate of  $\text{Al}(\text{HO})_3$ .

2.  $\text{KHO}$  or  $\text{NaHO}$  produces also a precipitate of  $\text{Al}(\text{HO})_3$ , soluble in acids, even in hot acetic acid and in excess of the reagent. This solution is not precipitated by  $\text{H}_2\text{S}$ , but is reprecipitated by  $\text{NH}_4\text{Cl}$ , or by adding  $(\text{NH}_4)\text{HO}$  after acidifying with  $\text{HCl}$ .

3.  $(\text{NH}_4)\text{HO}$  also precipitates  $\text{Al}(\text{HO})_3$ , soluble in a very large excess of the reagent, more difficultly soluble in presence of Salts of Ammonia.

4.  $\text{BaCO}_3$  produces a precipitate of  $\text{Al}(\text{OH})_3$  mixed with basic salt.

5.  $\text{Na}_2\text{HPO}_4$  precipitates aluminium phosphate, insoluble in  $\text{NH}_4(\text{OH})$  and in  $\text{NH}_4\text{Cl}$ , but soluble in  $\text{KHO}$  or  $\text{NaHO}$  and in acids. It does not, however, dissolve in hot Acetic Acid like  $\text{Al}(\text{OH})_3$ .

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6. Heated on charcoal in the blow-pipe flame and then moistened with  $\text{Co}(\text{NO}_3)_2$  and reheated, an infusible blue mass is obtained.

MANGANESE, Mn, 55. SOLUTION,  $\text{MnSO}_4$

1.  $(\text{NH}_4)_2\text{S}$  produces a flesh coloured precipitate of  $\text{MnS}$ , soluble in acids, even in Acetic Acid.

2.  $\text{KHO}$  or  $\text{NaHO}$  produces a dirty-white precipitate of  $\text{Mn}(\text{HO})_2$ , insoluble in excess of the reagent; the precipitate rapidly darkens in colour by absorption of Oxygen. The freshly-precipitated hydrate is dissolved by  $\text{NH}_4\text{Cl}$ , but the higher oxide is insoluble.

3.  $(\text{NH}_4)\text{HO}$  produces the same precipitate of  $\text{Mn}(\text{HO})_2$ , insoluble in excess of the reagent; but it gives no precipitate if the Manganese solution contain  $\text{NH}_4\text{Cl}$ . Such a solution on standing precipitates the dark-brown hydrate.

4.  $\text{Na}_2\text{CO}_3$  produces a white precipitate of  $\text{MnCO}_3$ , which darkens in colour by absorption of Oxygen.

5. If any Manganese solution (free from Chlorine) be treated with  $\text{PbO}_2$  and then boiled with  $\text{HNO}_3$ , it is converted into Permanganate, which is recognized by its pink colour as soon as the mixture has settled.

\*6. If any Manganese compound be fused on platinum foil with  $\text{Na}_2\text{CO}_3$ , and a trace of  $\text{KNO}_3$ , it is converted into  $\text{Na}_2\text{MnO}_4$ , recognized by its bright-green colour.

7. Fused with Borax in the oxidizing flame an amethyst-coloured bead is obtained, which becomes colourless in the reducing flame.

CHROMIUM, Cr, 52.1. SOLUTION,  $\text{Cr}_2(\text{SO}_4)_3$ .

1.  $(\text{NH}_4)_2\text{S}$  produces a bluish green precipitate of  $\text{Cr}(\text{HO})_3$ , insoluble in excess of the reagent, soluble in acids.

2.  $(\text{NH}_4)\text{HO}$  also precipitates the hydrate, soluble to some extent in excess, yielding a pink fluid, but on heating the precipitation is complete.

3.  $\text{KHO}$  or  $\text{NaHO}$  precipitates also  $\text{Cr}(\text{HO})_3$ , soluble however in excess, yielding a green or bluish-violet solution. On continued boiling or addition of  $\text{NH}_4\text{Cl}$  and heating, the hydrate is reprecipitated.

4.  $\text{BaCO}_3$  produces a precipitate of  $\text{Cr}(\text{HO})_3$  along with basic salt; the precipitation is not complete till the mixture has stood some time.

5. Fused with  $\text{Na}_2\text{CO}_3$  and  $\text{KNO}_3$  on platinum foil, yellow  $\text{Na}_2\text{CrO}_4$  is obtained.

6. Fused with Borax in either flame (but best in the reducing flame), green beads are obtained.

Characteristic reactions, colour of solutions and bead.

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TABLE O.—IRON GROUP (III)—SEPARATION OF IRON, NICKEL, COBALT, ALUMINIUM, ZINC, MANGANESE, AND CHROMIUM.

To the filtrate from the Sulphides of the Cu and As groups add  $(\text{NH}_4)_2\text{CO}_3$  (till alkaline) +  $(\text{NH}_4)_2\text{S}$  +  $(\text{NH}_4)_2\text{S}_2\text{O}_8$ . Warm the mixture gently in a small flask and shake for some time. Filter (preferably by the Bunsen pump). Wash well with  $\text{H}_2\text{O}$ , containing  $(\text{NH}_4)_2\text{S}_2\text{O}_8$ , and finally once with  $\text{H}_2\text{O}$  alone. The filtrate is often brown in presence of Ni. Treat the precipitate with cold dilute HCl, and filter.

Nis and CoS.		Cr, Al, Fe, Zn, and Mn.	
TEST FOR Co by Borax Bead.		(Green or violet if Cr be present. Boil down a portion and test for Cr by Borax Bead. Adopt Method I. if absent, Method II. if present.)	
Dissolve the black residue in HCl and $\text{KClO}_3$ . Boil down just to dryness, dilute with $\text{H}_2\text{O}$ , and add KCN in excess, then a drop of Acetic Acid, boil for a few minutes, add $\text{NaClO}_4$ in excess, and boil precipitate indicates Nickel.	Method I.—Cr is Absent. Boil down with a little $\text{KClO}_3$ till it smells of Cl. neutral or slightly acid; allow to become perfectly cold. Add excess of $\text{BaCO}_3$ , place in a flask, cork up and shake well, allow to stand till clear. Filter	Method II.—Cr is Present. Boil down with a little $\text{KClO}_3$ till it smells of Cl. neutral or slightly acid; allow to become perfectly cold. Add excess of $\text{BaCO}_3$ , place in a flask, cork up and shake well, allow to stand till clear. Filter	FILTRATE.
RESIDUE, $\text{Fe}(\text{HO})_3$ , $\text{Mn}(\text{HO})_2$ .	RESIDUE, $\text{Fe}(\text{HO})_3$ , $\text{Cr}(\text{HO})_3$ , $\text{Al}(\text{HO})_3$ (also excess of $\text{BaCO}_3$ ).	RESIDUE, $\text{Fe}(\text{HO})_3$ , $\text{Cr}(\text{HO})_3$ , $\text{Al}(\text{HO})_3$ (also excess of $\text{BaCO}_3$ ).	FILTRATE.
Dissolve in HCl. Test with $\text{K}_4\text{Fe}(\text{CN})_6$ . Blue precipitate indicates Iron. To ascertain whether iron is present as ferric salt, the original solution must be treated with $\text{K}_4\text{Fe}(\text{CN})_6$ , and $\text{K}_3\text{Fe}(\text{CN})_6$ .	Divide into two parts. 1. Add $\text{H}_2\text{S}$ or $(\text{NH}_4)_2\text{S}$ . A white precipitate indicates Zinc. Confirm by flame-reaction. 2. Add HCl till acid, then $(\text{NH}_4)_2\text{HO}$ till alkaline. A white precipitate indicates Aluminium. Confirm by flame reaction.	Wash well, boil with pure $\text{NaHO}$ , and filter; add HCl to the filtrate, and then $(\text{NH}_4)_2\text{HO}$ till alkaline. A white precipitate indicates Aluminium. Confirm by flame-reaction. Fuse the residue with water, and filter.	Zn, Mn. Precipitate the Ba present with $\text{H}_2\text{SO}_4$ in the hot solution. Boil well, and filter; add $\text{NaHO}$ . Precipitate indicates Manganese. Confirm by fusing with $\text{Na}_2\text{CO}_3$ and $\text{KNO}_3$ on platinum foil. To the filtrate from $(\text{NH}_4)_2\text{HO}$ , add $(\text{NH}_4)_2\text{S}_2\text{O}_8$ . A white precipitate indicates Zinc. Confirm by flame-reaction.
RESIDUE, $\text{Fe}(\text{HO})_3$ .	RESIDUE, $\text{Fe}_2(\text{HO})_6$ .	RESIDUE, $\text{Fe}_2(\text{HO})_6$ .	FILTRATE.
Boil down and ignite, to expel minimum amount of $\text{H}_2\text{O}$ . Fuse with $\text{NaHO}$ and $\text{KNO}_3$ . A green residue indicates Magnesia or ferric salt, the original solution must be treated with $\text{K}_4\text{Fe}(\text{CN})_6$ , and $\text{K}_3\text{Fe}(\text{CN})_6$ .	Dissolve in HCl, and test with $\text{K}_4\text{Fe}(\text{CN})_6$ . A blue precipitate indicates Iron.	Dissolve in HCl, and test with $\text{K}_4\text{Fe}(\text{CN})_6$ . A blue precipitate indicates Iron.	FILTRATE.
Boil down and ignite, to expel minimum amount of $\text{H}_2\text{O}$ . Fuse with $\text{NaHO}$ and $\text{KNO}_3$ . A green residue indicates Magnesia or ferric salt, the original solution must be treated with $\text{K}_4\text{Fe}(\text{CN})_6$ , and $\text{K}_3\text{Fe}(\text{CN})_6$ .	Yellow in colour, Acidify with Acetic Acid; add Lead Acetate. A bright yellow precipitate indicates Chromium.	Yellow in colour, Acidify with Acetic Acid; add Lead Acetate. A bright yellow precipitate indicates Chromium.	FILTRATE.
Another Method.—Boil filtrate from Cu group to expel $\text{H}_2\text{S}$ , then boil with $\text{KClO}_3$ , add $\text{NH}_4\text{Cl}$ , and then $(\text{NH}_4)_2\text{HO}$ till alkaline. Filter.	Another Method.—Boil filtrate from Cu group to expel $\text{H}_2\text{S}$ , then boil with $\text{KClO}_3$ , add $\text{NH}_4\text{Cl}$ , and then $(\text{NH}_4)_2\text{HO}$ till alkaline. Filter.	Another Method.—Boil filtrate from Cu group to expel $\text{H}_2\text{S}$ , then boil with $\text{KClO}_3$ , add $\text{NH}_4\text{Cl}$ , and then $(\text{NH}_4)_2\text{HO}$ till alkaline. Filter.	FILTRATE.
Test washed ppt. for Cr by Borax Bead, and for Fe with $\text{K}_4\text{Fe}(\text{CN})_6$ . Treat residue with warm $\text{NaHO}$ , and test for Al in filtrate as in Method I. 2.	Test washed ppt. for Cr by Borax Bead, and for Fe with $\text{K}_4\text{Fe}(\text{CN})_6$ . Treat residue with warm $\text{NaHO}$ , and test for Al in filtrate as in Method I. 2.	Test washed ppt. for Cr by Borax Bead, and for Fe with $\text{K}_4\text{Fe}(\text{CN})_6$ . Treat residue with warm $\text{NaHO}$ , and test for Al in filtrate as in Method I. 2.	FILTRATE.

\*  $\text{NaHO}$  and Bromine Water may be used instead of  $\text{NaClO}$ .

† When this method is used the filtrate cannot be afterwards tested for Potassium.

Warm  $\text{NaHO}$ , and test for Al in  
filtrate as in Method I. 2.  
add  $\text{NaHO}$  till Alkaline, and separ-  
ate Mn and Zn as in Method II.

† When this method is used the filtrate cannot be afterwards tested for Potassium.

\*  $\text{NaHO}$  and Bromine Water may be used instead of  $\text{NaClO}$ .

Reactions of the Metals of the Barium Group—(Group IV).

Metals whose carbonates are insoluble in water, and whose solutions are precipitated on the addition of  $(\text{NH}_4)_2\text{CO}_3$ . As, however, the carbonates are soluble in acids, the solution, if acid, must be neutralized by addition of  $(\text{NH}_4)\text{HO}$ .

Barium, Strontium, Calcium.

BARIUM, Ba, 137. SOLUTION,  $\text{BaCl}_2$ .

1.  $(\text{NH}_4)_2\text{CO}_3$  precipitates white  $\text{BaCO}_3$ , soluble in acids, and to a slight extent in  $\text{NH}_4\text{Cl}$ .

2.  $\text{K}_2\text{CO}_3$  or  $\text{Na}_2\text{CO}_3$  precipitates also  $\text{BaCO}_3$ , insoluble in excess of either reagent.

\*3.  $\text{H}_2\text{SO}_4$  or any soluble sulphate, even in dilute solutions, precipitates heavy white  $\text{BaSO}_4$ , insoluble in Acids, Alkalies or Salts of Ammonium.

4.  $\text{CaSO}_4$  or  $\text{SrSO}_4$  precipitates immediately white  $\text{BaSO}_4$ .

5.  $\text{H}_2\text{SiF}_6$  precipitates white  $\text{BaSiF}_6$ .

6.  $\text{C}_2(\text{NH}_4)_2\text{O}_4$  precipitates white  $\text{C}_2\text{BaO}_4$ , soluble in  $\text{HCl}$  and in  $\text{HNO}_3$ .

7.  $\text{K}_2\text{CrO}_4$  precipitates yellow  $\text{BaCrO}_4$ , insoluble in  $\text{C}_2\text{H}_4\text{O}_2$ , but soluble in  $\text{HCl}$  and  $\text{HNO}_3$ .

\*8. Heated in the lamp flame, a green colouration is produced, especially on moistening the salt with  $\text{HCl}$ .

Write all equations.

STRONTIUM, Sr, 87.5. SOLUTION,  $\text{Sr}(\text{NO}_3)_2$ .

1.  $(\text{NH}_4)_2\text{CO}_3$  or  $\text{K}_2\text{CO}_3$  precipitates white  $\text{SrCO}_3$ , soluble in acids, but less soluble in  $\text{NH}_4\text{Cl}$  than  $\text{BaCO}_3$ .

2.  $\text{H}_2\text{SO}_4$  precipitates white  $\text{SrSO}_4$ , much less insoluble in  $\text{H}_2\text{O}$  than  $\text{BaSO}_4$ ; it therefore precipitates from dilute solutions only on standing or warming.  $\text{SrSO}_4$  is slightly soluble in  $\text{HCl}$ .

\*3.  $\text{CaSO}_4$ , after standing some time, precipitates white  $\text{SrSO}_4$ .

4.  $\text{H}_2\text{SiF}_6$  does not precipitate strontium solutions.

5.  $\text{C}_2(\text{NH}_4)_2\text{O}_4$  precipitates white  $\text{C}_2\text{SrO}_4$ , soluble in  $\text{HCl}$  and in  $\text{HNO}_3$ , also to a slight extent in  $\text{NH}_4\text{Cl}$ , but very sparingly in  $\text{C}_2\text{H}_4\text{O}_2$ .

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6.  $K_2CrO_4$ , only in concentrated solutions, precipitates yellow  $SrCrO_4$ , soluble in  $C_2H_4O_2$ .

\*7. Heated in the lamp flame, a crimson colouration is produced, especially on moistening the salt with HCl.

CALCIUM,  $Ca^{II}$ , 40. SOLUTION,  $CaCl_2$ .

1.  $(NH_4)_2CO_3$  or  $K_2CO_3$  precipitates white  $CaCO_3$ , which becomes crystalline on heating.

2.  $H_2SO_4$  precipitates from strong solutions of calcium salts  $CaSO_4$  as a white precipitate, which dissolves in a large excess of water, and also in acids.

3.  $CaSO_4$  produces no precipitate.

4.  $H_2SiF_6$  produces no precipitate.

\*5.  $C_2(NH_4)_2O_4$ , even in dilute solutions, precipitates white  $C_2CaO_4$ , soluble in HCl or  $HNO_3$ , but insoluble in  $C_2H_2O_4$  or in  $C_2H_4O_2$ .

\*6. Heated in the lamp flame, a dull-red colouration is produced, especially on moistening the salt with HCl. This reaction is imperceptible in presence of Ba or Sr salts.

Tabulate the chemical properties of Calcium Barium and Strontium.

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TABLE E.—CALCIUM GROUP (IV). SEPARATION OF BARIUM, STRONTIUM, AND CALCIUM.

Heat filtrate from iron group, add to the hot solution  $\text{NH}_4\text{Cl}$  and  $(\text{NH}_4)_2\text{CO}_3$ , and filter. Wash precipitate with hot  $\text{H}_2\text{O}$ , dissolve in  $\text{HCl}$ , and add to a portion  $\text{CaSO}_4$  solution. An immediate precipitate indicates **Barium**; a precipitate after some time indicates **Strontium**, or a dilute Barium solution. (Test another portion with  $\text{SrSO}_4$  for Ba.) To another portion of the solution in  $\text{HCl}$  add  $\text{H}_2\text{SO}_4$ , and boil to remove Ba and Sr. Filter. Neutralize filtrate with  $(\text{NH}_4)_2\text{HO}$ , and add  $\text{C}_2(\text{NH}_4)_2\text{O}_4$ . An immediate precipitate indicates **Calcium**.

TO TEST FOR STRONTIUM.

I. Ba PRESENT. Ca ABSENT.	II. Ba ABSENT. Ca PRESENT.	III. Ba AND Ca PRESENT
Dissolve the carbonate in $\text{HCl}$ , and evaporate to dryness. Treat the residue with strong alcohol. Filter. RESIDUE. <span style="float:right">FILTRATE.</span>	Dissolve as before, and precipitate with $\text{H}_2\text{SO}_4$ . Filter, and wash well. RESIDUE. <span style="float:right">FILTRATE.</span>	Add $\text{H}_2\text{SO}_4$ to the $\text{HCl}$ solution (diluted to prevent precipitation of Calcium), and filter. RESIDUE. <span style="float:right">FILTRATE.</span>
$\text{BaCl}_2$ . Confirm by flame test. Green colouration indicates <b>Barium</b> . Confirm by lighting the alcoholic solution. Crimson colouration indicates <b>Strontium</b> . Sr. Confirm by lighting the alcoholic solution. Crimson colouration indicates <b>Strontium</b> . Strontium.	$\text{SrSO}_4$ . If small, burn the filter in the reducing gas flame to convert $\text{SrSO}_4$ into SrS: moisten with $\text{HCl}$ , and test in the lamp flame. Crimson colouration indicates <b>Strontium</b> . Ca. Neutralize the solution $(\text{NH}_4)_2\text{HO}$ , and test with $\text{C}_2(\text{NH}_4)_2\text{O}_4$ . White precipitate indicates <b>Calcium</b> .	$\text{Ba}_2\text{SO}_4$ , $\text{SrSO}_4$ . Boil in a beaker with a little water, together with a mixture of three parts $(\text{NH}_4)_2\text{O}_4$ . White $\text{K}_2\text{SO}_4$ and one part $\text{K}_2\text{CO}_3$ . Filter and treat residue with $\text{HNO}_3$ . The $\text{SrSO}_4$ is dissolved, and the $\text{BaSO}_4$ left undissolved. (Traces of Ca may be found with the Sr.) Ca. Neutralize the solution with $\text{NH}_4(\text{HO})$ , and test with $\text{C}_2(\text{NH}_4)_2\text{O}_4$ . White precipitate indicates <b>Calcium</b> .
ANOTHER METHOD. Dissolve the $\text{HNO}_3$ , and evaporate to dryness. Treat with strong alcohol. Filter. RESIDUE. <span style="float:right">FILTRATE.</span> $\text{Sr}(\text{NO}_3)_2$ . Confirm as above. <span style="float:right">Ca.</span> Confirm as above. <span style="float:right">Confirm as above.</span>		ANOTHER METHOD. Dissolve the carbonates in $\text{C}_2\text{H}_4\text{O}_3$ , and precipitate the Ba with $\text{K}_2\text{CrO}_4$ . Filter. Precipitate the Sr and Ca by $(\text{NH}_4)_2\text{CO}_3$ , and proceed as in Method II. (Ba Absent, Ca Present).

Confirm as above. | Ca Present).

Confirm as above.

## Reactions of the Metals of the Potassium Group.

(Group V.)

Metals whose solutions are unprecipitated by the preceding group reagents, and having no common precipitant. They are therefore recognized by individual tests.

## Magnesium, Potassium, Ammonium, Sodium.

MAGNESIUM, Mg, 24. SOLUTION,  $\text{MgSO}_4$ .

1.  $(\text{NH}_4)\text{HO}$  and  $(\text{NH}_4)_2\text{CO}_3$  give no precipitates in the presence of salts of ammonium.

\*2.  $\text{Na}_2\text{HPO}_4$  in presence of  $(\text{NH}_4)\text{HO}$  and  $(\text{NH}_4)\text{Cl}$  a crystalline white precipitate of  $\text{MgNH}_4\text{PO}_4$ . The precipitation is slow from dilute solutions, but may be hastened by stirring with a glass rod and warming. Precipitate soluble in dilute mineral acids and in  $\text{C}_2\text{H}_4\text{O}_2$ , almost insoluble in dilute solution of  $(\text{NH}_4)\text{HO}$ .

3.  $\text{H}_2\text{SO}_4$ ,  $\text{H}_2\text{SiF}_6$ , and  $\text{C}_2(\text{NH}_4)_2\text{O}_4$  give no precipitates.

\*4. Heated on charcoal in the blow-pipe flame, and then moistened with  $\text{CoCl}_2$  or  $\text{Co}(\text{NO}_3)_2$  and reheated, gives a pink mass.

POTASSIUM, K, 39.1. SOLUTION,  $\text{KCl}$ .

1.  $\text{PtCl}_2$ , except in dilute solutions, gives a crystalline yellow precipitate of  $2\text{KCl} + \text{PtCl}_4$ . The precipitation is hastened by stirring or the addition of alcohol.

2.  $\text{H}_2\text{Tr}$  or  $\text{NaHTr}$  precipitates white crystalline  $\text{KHTr}$  from concentrated solutions.

3.  $\text{H}_2\text{SiF}_6$  precipitates white gelatinous  $\text{K}_2\text{SiF}_6$ .

\*4. Heated on platinum wire, potassium compounds colour the flame violet, appearing reddish-violet through blue glass.

AMMONIUM,  $\text{NH}_4$ , 18. SOLUTION,  $\text{NH}_4\text{Cl}$ .

1.  $\text{PtCl}_4$  gives a crystalline yellow precipitate of  $2\text{NH}_4\text{Cl} + \text{PtCl}_4$ , except in dilute solutions. Precipitate insoluble in alcohol and ether. On ignition, precipitate leaves a residue of spongy platinum.

2.  $\text{NaHTr}$  or  $\text{H}_2\text{Tr}$  give in strong solutions a white precipitate of  $(\text{NH}_4)\text{HTr}$ .

3.  $\text{H}_2\text{SiF}_6$  gives no precipitate.

\*4. Nessler's solution gives a brown precipitate, or in any dilute solutions a yellow colouration.

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\*5. Heated with NaHO or KHO, compounds of Ammonium evolve Ammonia gas, recognized by its odour, alkaline reaction, and fuming with HCl.

\*6. Heated on platinum foil, all compounds of Ammonia volatilize completely.

### SODIUM, Na, 23. SOLUTION, NaCl.

1.  $\text{PtCl}_4$ , NaHTr, and  $\text{H}_2\text{Tr}$  gives no precipitates.
2.  $\text{H}_2\text{SiF}_6$  gives precipitate of white gelatinous  $\text{Na}_2\text{SiF}_6$ .
- \*3. The Salts of Sodium being almost without an exception soluble in water, the flame test alone serves to distinguish the metal. Heated on platinum foil or wire in non-luminous flame, an intense yellow colour is produced, not seen when viewed through blue glass.

TABLE F.—GROUP V.

#### Separation of Magnesium, Potassium, Sodium and Ammonium.

The filtrate from the Barium Group is concentrated by evaporation, and a portion ignited on Platinum Foil. If no residue is left on ignition, Mg, K, and Na are absent.

Detection of $\text{NH}_4$	Detection of Mg.	Detection of K and Na.	
The original substance or solution is heated with NaHO in a test tube. Presence of Ammonium shown by smell, by the white fumes with HCl, and by its action on red litmus paper.	To a portion of the concentrated cold solution add $(\text{NH}_4)\text{HO}$ and $\text{Na}_2\text{HPO}_4$ . White Crystalline Precipitate denotes Magnesium.	(1) Mg. being absent.	(2) Mg. being present.
		Evaporate another portion of the solution to dryness, ignite residue, dissolve in a small quantity of water, filter if required, and add to the clear liquid $\text{PtCl}_4$ , evaporate nearly to dryness, and add alcohol. Yellow Precipitate indicates Potassium.	Evaporate the solution to dryness, ignite residue, dissolve in water, and add Baryta Water until the solution has an alkaline reaction; boil; filter. To filtrate add $(\text{NH}_4)_2\text{CO}_3$ , heat, filter, evaporate to dryness, and test the residue for K and Na.
	<b>To Detect Na.</b> Evaporate alcoholic solution (which must have a yellow colour, showing that excess of $\text{PtCl}_4$ has been added) nearly to dryness, add a grain or two of sugar, and ignite residue. Exhaust with water, filter, evaporate to dryness; and if a residue be left, test it by flame reaction for Na. Yellow colouration indicates Sodium.		

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TABLE SHOWING THE SOLUBILITY IN WATER AND ACIDS OF THE COMMON METALLIC SALTS.

	Acetate.	Arsenate.	Arsenite.	Borate.	Bromide.	Carbonate.	Chlorate.	Chloride.	Chromate.	Cyanide.	Ferricyanide.	Ferrocyanide.	Fluoride.	Iodide.	Nitrate.	Oxalate.	Oxide.	Phosphate.	Silicate.	Sulphate.	Sulphide.
Ag .....	s	i	i	i	I	i	S	I	i	i	I	I	S	i	S	i	i	i		s	i
Pb .....	S	i	i	i	sI	i	S	sI	i	i	s	i	s	i	S	i	i	i	i	I	i
Hg <sub>2</sub> .....	s	i	i		i	i	S	i	i	i	i	i	S	i	S	i	i	i		s	i
Hg .....	S	i	i		s	i	S	S	s	S			S	i	Si	i	i	i		Si	i
Bi .....	S	i		i	Si	i	S	Si	i	s		I	s	Si	Si	i	i	i		Si	i
Cu .....	S	i	i	i	S	i	S	S	s	i	I	I	s	Si	S	i	i	i	i	S	i
Cd .....	S	i	s	S	i	S	S	S	i	i			s	S	S	i	i	i	i	S	i
Sn <sup>ii</sup> .....	S	i	i	i	S			Si	i		i	i	S	S	Si	i	i	i		S	i
Sn <sup>iv</sup> .....	S	i			S			S				i	S	Si	Si	S	i	i		S	i
Sb .....	S	i	i		Si			Si	i				S	Si		i	i	i		Si	i
Fe <sup>ii</sup> .....	S	i	i	i	S	i	S	S		i	I	I	s	S	S	i	i	i	i	S	i
Fe <sup>iv</sup> .....	S	i	i	i	S		S	S	s		S	I	s	S	S	s	i	i	i	S	i
Cr .....	S	i		i	S		S	S	i	i			s	S	S	S	i	i	i	S	i
Al .....	S	i		i	S		S	S					i		S	i	i	i		S	i
Mn .....	S	i	i	i	S	i	S	S	s	i	I	i	i	S	S	i	i	i	i	S	i
Zn .....	S	i		i	S	i	S	S	S	i	i	i	s	S	S	i	i	i	i	S	i
Co .....	S	i	i	i	S	i	S	S	i	i	I	I	s	S	S	i	i	i	i	S	i
Ni .....	S	i	i	i	S	i	S	S	i	i	I	I	s	S	S	i	i	i	i	S	i
Ba .....	S	i	s	i	S	i	S	S	i	s	s	s	i	S	S	i	i	i	i	I	S
Sr .....	S	i	s	i	S	i	S	S	s	S		S	i	S	S	i	i	i	i	I	S
Ca .....	S	i	i	i	S	i	S	S	s	S	S	S	i	S	S	i	s	i	i	sI	S
Mg .....	S	i	i	s	S	i	S	S	S	S	S	S	i	S	S	s	i	i	i	S	S
K .....	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S
NH <sub>4</sub> .....	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Na .....	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S

S, soluble in water; s, sparingly soluble in water; i, insoluble in water, but made soluble by acids; I, insoluble in water or acids; sI, sparingly soluble in water, not transposed by acids; Si, soluble in acidulated water.



## SECTION IV.

### REACTIONS OF THE ACIDS.

#### Grouping of the Acids.

The acids do not admit of being grouped with the same precision as the bases, but they can be approximately classified by means of certain group reagents. They are divided into two great classes, Inorganic and Organic Acids. These are readily distinguished by the action of heat. Salts of Inorganic Acids when heated to redness are not charred; salts of Organic Acids, with the exception of acetic and formic acids, are at once charred, owing to decomposition and consequent separation of carbon.

#### Grouping of the Inorganic Acids.

##### GROUP I. (SULPHURIC ACID GROUP).

Group reagent,  $\text{BaCl}_2$  in presence of  $\text{HCl}$ .

##### Sulphuric Acid, Hydrofluosilicic Acid.

The acids of this group are precipitated by  $\text{BaCl}_2$ , and the precipitate is not dissolved on addition of  $\text{HCl}$ .

##### GROUP II. (PHOSPHORIC ACID GROUP).

Group reagent,  $\text{BaCl}_2$ .

Phosphoric, Boric, Oxalic, Hydrofluoric, Carbonic, Silicic, Sulphurous, Hyposulphurous, Arsenious, Arsenic, Iodic, and Chromic Acids.

The acids of this group are precipitated in *neutral solutions* by  $\text{BaCl}_2$ .

##### GROUP III. (HYDROCHLORIC ACID GROUP.)

Group reagent,  $\text{AgNO}_3$ .

Hydrochloric, Hydrobromic, Hydriodic, Hydrocyanic, Hypochlorous, Nitrous, and Hydrosulphuric Acids.

The acids of this group are precipitated by  $\text{AgNO}_3$ , and *not* by  $\text{BaCl}_2$ .

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## GROUP IV. (NITRIC ACID GROUP).

## Nitric, Chloric, and Perchloric Acids.

These acids are not precipitated by any reagent, as all their salts are soluble in water.

## Reactions of the Inorganic Acids belonging to Group 1.

1. Acids precipitated by  $\text{BaCl}_2$  in presence of  $\text{HCl}$ .

## Sulphuric Acid, Hydroflu-silicic Acid.

SULPHURIC ACID.  $\text{H}_2\text{SO}_4$ , c.w. 98.

1.  $\text{BaCl}_2$  produces a white precipitate of  $\text{BaSO}_4$ , insoluble in  $\text{HCl}$  or  $\text{HNO}_3$ . In very dilute solutions the precipitation is not immediate, but on standing the solution becomes clouded, and ultimately the precipitate subsides.

\*2.  $\text{Pb}(\text{NO}_3)_2$  produces a heavy white precipitate of  $\text{PbSO}_4$ , soluble in  $\text{NaHO}$ , and in boiling  $\text{HCl}$  (on allowing this solution to cool,  $\text{PbCl}_2$  crystallizes out.)

\*3. Fused on charcoal with  $\text{Na}_2\text{CO}_3$  in the reducing flame of the blow-pipe, a sulphide is produced. If the fused mass be moistened with  $\text{HCl}$ , the odour of  $\text{H}_2\text{S}$  is at once perceptible; or if it be placed on a bright piece of silver and moistened with water, a black stain of  $\text{Ag}_2\text{S}$  is produced. As the latter reaction is very delicate, care must be taken to use  $\text{Na}_2\text{CO}_3$  perfectly free from  $\text{Na}_2\text{SO}_4$ , and it must be borne in mind that the other sulphur acids give the same reaction when heated with  $\text{Na}_2\text{CO}_3$ .

2. HYDROFLUO-SILICIC ACID.  $\text{H}_2\text{SiF}_6$ , c.w. 144.

1.  $\text{BaCl}_2$  produces a crystalline precipitate of  $\text{BaSiF}_6$ , insoluble in  $\text{HCl}$ .

2.  $\text{KCl}$  produces a gelatinous precipitate of  $\text{K}_2\text{SiF}_6$ .

3. Heated with Sulphuric Acid in a platinum or leaden crucible covered with a watch-glass, the latter is etched owing to the disengagement of  $\text{HF}$ .

## Reactions of the Acids belonging to Group II.

3. Acids precipitated by  $\text{BaCl}_2$  in neutral solutions.

Phosphoric, Boric, Oxalic, Hydrofluoric, Carbonic, Silicic, Sulphurous, Hyposulphurous, Arsenious, Arsenic, Iodic, and Chromic Acids.

PHOSPHORIC ACID.  $\text{H}_3\text{PO}_4$ , c.w. 98. (ORTHO-PHOSPHORIC ACID.)

1.  $\text{BaCl}_2$  produces a white precipitate of  $\text{BaHPO}_4$ , readily soluble in  $\text{HNO}_3$  or  $\text{HCl}$ , but with difficulty in  $\text{NH}_4\text{Cl}$ .

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2.  $MgSO_4$  along with  $(NH_4)HO$  and  $NH_4Cl$ , produces a white crystalline precipitate of  $Mg(NH_4)PO_4 + 6H_2O$ , insoluble in  $(NH_4)HO$ , but soluble in  $HCl$ ,  $HNO_3$ , and Acetic Acid. In dilute solutions the precipitation does not take place till after the lapse of some time, but is promoted by stirring and gentle warming.

3.  $AgNO_3$  produces a yellow precipitate of  $Ag_3PO_4$ , soluble in  $HNO_3$ , and also in  $(NH_4)HO$ .

4. Lead acetate produces a white precipitate of  $Pb_3(PO_4)_2$ , soluble in  $HNO_3$ , but almost insoluble in Acetic Acid.

5.  $Fe_2Cl_6$ , in presence of excess of Sodium Acetate, produces a yellowish precipitate of  $FePO_4$ , soluble in  $HCl$ , and in excess of  $Fe_2Cl_6$ , which ought therefore to be added drop by drop.

\*6. Ammonium molybdate produces in solutions made acid with  $HNO_3$  a yellow colouration and ultimately a precipitate. The reaction is hastened by warming the mixture.

The following acids of phosphorus are distinguished from each other and from ortho phosphoric acid by the following reactions:—

*Pyrophosphoric Acid.*  $H_4P_2O_7$ , c.w. 178.

1.  $AgNO_3$  produces a white precipitate of  $Ag_4P_2O_7$ , soluble in  $HNO_3$ , and in  $(NH_4)HO$ .

2. Albumen gives no precipitate.

*Metaphosphoric Acid.*  $HPO_3$ , c.w. 80.

1.  $AgNO_3$  produces a white gelatinous precipitate of  $AgPO_3$ .

2. Albumen produces a flocculent white precipitate when added to metaphosphoric acid, and the same precipitate when added to a solution of a metaphosphate acidified with acetic acid.

3.  $MgSO_4 + NH_4Cl + (NH_4)HO$  produces no precipitate.

Tabulate your results.

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\*If the test be applied to an acid solution of a phosphate insoluble in water (e.g.  $Ca_3(PO_4)_2$  in  $HCl$ , the free acid is first nearly neutralized with  $(NH_4)HO$ , sodium acetate next added, and then  $Fe_2Cl_6$ ; after this the mixture is boiled. The precipitate, which is of a reddish-brown colour, contains all the iron and phosphoric acid: the filtrate contains the base. The phosphoric acid can easily be separated from the iron, and obtained as a soluble ammonium salt by treating the ferric phosphate with  $(NH_4)_2S$ .

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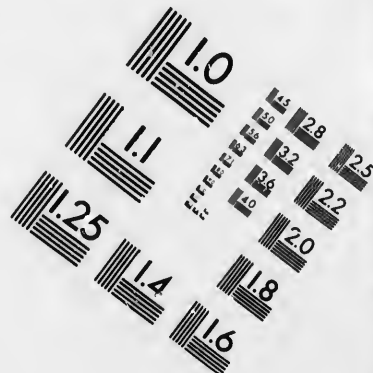
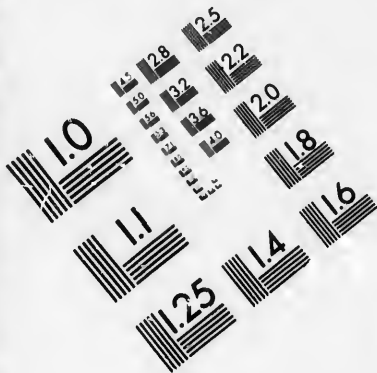
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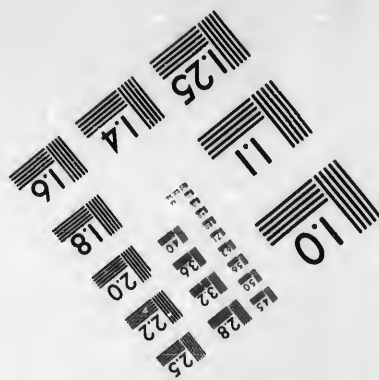
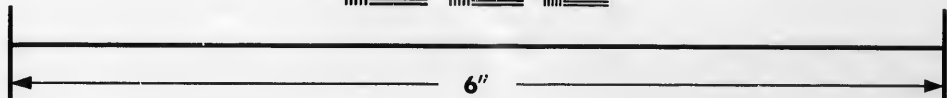
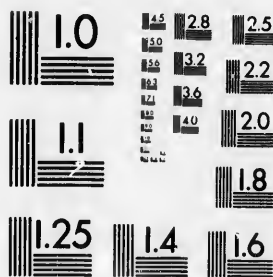
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4. BORIC ACID.  $B(OH)_3$ , c.w. 62.

1.  $BaCl_2$  produces a white precipitate of  $Ba(BO_2)_2$ , soluble in acids.

2.  $AgNO_3$  produces in strong solutions a yellowish white precipitate. In dilute solutions  $Ag_2O$  is precipitated.

3.  $H_2SO_4$  or  $HCl$ , added to hot concentrated solutions of alkaline borates, produces on cooling a crystalline precipitate of  $B(OH)_3$ .

\*4. If alcohol containing free boric acid be kindled, it burns with a green flame, best seen on stirring the mixture. Borates may be examined in this way by first adding strong  $H_2SO_4$ , to liberate the  $B(OH)_3$ .

5. If the solution of a borate be made distinctly acid with  $HCl$ , and turmeric paper dipped into it, the latter on gentle warming acquires a brown tint, which is turned blue by caustic soda.

6. Moistened with  $H_2SO_4$  and heated in the lamp flame, a green colouration is produced.

5. OXALIC ACID.  $C_2H_2O_4$ ,  $\begin{matrix} CO_2H \\ CO_2H \end{matrix}$  c.w. 90.

1.  $BaCl_2$  produces in neutral solutions a white precipitate of  $C_2O_4Ba$ , soluble in  $HNO_3$ , in  $HCl$ , and in  $C_2H_2O_4$ .

2.  $AgNO_3$  produces a white precipitate of  $C_2O_4Ag_2$ , soluble in  $HNO_3$ , and in  $(NH_4)HO$ .

3.  $CaCl_2$  produces even in dilute solutions a white precipitate of  $C_2O_4Ca$ , soluble in  $HNO_3$ , and in  $HCl$ , but nearly insoluble in  $(NH_4)HO$ , and in acetic acid. On igniting  $C_2O_4Ca$ , a white residue of  $CaCO_3$  is left, which effervesces on the addition of an acid.

\*4. Heated with strong  $H_2SO_4$ , effervescence takes place from the escape of a mixture of  $CO$  and  $CO_2$ , and the former may be kindled at the mouth of the test tube, and will burn with a pale blue flame. *No blackening* of the mixture occurs as in the case of organic acids, which yield  $CO$  on heating with  $H_2SO_4$ .

6. HYDROFLUORIC ACID.  $HF$ , c.w. 20.

1.  $BaCl_2$  produces a white precipitate of  $BaF_2$ , soluble in  $HCl$ , and sparingly in  $NH_4Cl$ .

2.  $CaCl_2$  produces a gelatinous and almost transparent precipitate of  $CaF_2$ , difficult to discern in the fluid, but made more appar-

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ent on addition of  $(\text{NH}_4)\text{HO}$ . The precipitate is very difficultly soluble in  $\text{HCl}$ , even on boiling, and is nearly insoluble in Acetic Acid.

\* 3. Heated with  $\text{H}_2\text{SO}_4$ , all fluorides are decomposed with evolution of  $\text{HF}$ , which is recognized by its power of etching glass. A *very characteristic* oily appearance is noticed whenever a fluoride is warmed with  $\text{H}_2\text{SO}_4$ , in a test tube.

The etching is best done by placing the fluoride in a platinum crucible, or small leaden cup, along with strong  $\text{H}_2\text{SO}_4$ , and covering the mouth with a waxed watchglass, convex side downwards, on which a few scratches have been made with a needle. The concave side of the watchglass is filled with water to prevent the wax on the other side from melting, and the crucible or cup is then gently heated. On removing the glass and melting off the wax by gentle warming, the glass will be found etched at the unprotected parts.\*

4. Heated with a mixture of Borax and  $\text{HKSO}_4$ , on a loop of platinum wire in the non-luminous gas flame,  $\text{BF}_3$  is produced, which momentarily colours the flame green.

#### 7. CARBONIC ACID. $\text{H}_2\text{CO}_3$ , $\text{H}_2\text{O} + \text{CO}_2$ or $\text{CO} \begin{matrix} \text{OH} \\ \text{OH} \end{matrix}$

1.  $\text{BaCl}_2$  produces in neutral solutions a white precipitate of  $\text{BaCO}_3$ , soluble in acids with effervescence.

\*2. Treated with dilute  $\text{HCl}$ , all carbonates at once evolve  $\text{CO}_2$  with effervescence, and if this gas be conducted into lime-water it produces a turbidity from formation of  $\text{CaCO}_3$ . (The experiment may be conveniently performed by placing the carbonate and dilute acid in one test tube, and the lime-water in another. As soon as the  $\text{CO}_2$  has collected, it may be decanted into the lime-water tube—care being taken to prevent any liquid from being decanted with it—and on shaking the latter the lime-water will become turbid.)

#### 8. SILICIC ACID. $\text{Si}(\text{HO})_4$ , cw. 96.

1.  $\text{BaCl}_2$  produces a white precipitate of  $\text{SiBa}_2\text{O}_4$ , which is decomposed on addition of  $\text{HCl}$ , and  $\text{Si}(\text{HO})_4$  separates out as a gelatinous precipitate.

2.  $\text{HCl}$ , added drop by drop to a strong solution of a silicate,

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\* If the fluoride contain much silica,  $\text{SiF}_4$  is evolved instead of  $\text{HF}$ , and is detected by heating the substance with  $\text{H}_2\text{SO}_4$  in a test tube, and leading the evolved gas into water. Silica will separate out in flocculent tufts, and  $\text{H}_2\text{SiF}_6$  will be found in solution.

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produces a gelatinous precipitate of  $\text{Si}(\text{HO})_4$ , but if added to a dilute solution or in large excess, no precipitate is obtained until the mixture has been evaporated to dryness and ignited, when  $\text{SiO}_2$  separates out, and this is not re-dissolved on addition of  $\text{HCl}$ .

3. Fused with  $\text{Na}_2\text{CO}_3$  in a loop of platinum wire in the non-luminous gas flame, effervescence occurs from the disengagement of  $\text{CO}_2$ , and the bead is transparent on cooling, unless the  $\text{Na}_2\text{CO}_3$  be in excess.

\*4. Fused with Microcosmic Salt on a loop of platinum wire in the non-luminous gas flame, solution does not take place, but the silica floats about on the bead undissolved.

9. The remaining six acids of this group are precipitated or decomposed by one or other of the group reagents for *bases*, and are therefore precipitated in the course of examination for bases, or expelled on the addition of  $\text{HCl}$ . The action of the *base* group-reagents is as follows:—

Ag Group.	{	$\text{H}_2\text{SO}_3$ decomposed by $\text{HCl}$ , with evolution of $\text{SO}_2$ . $\text{H}_2\text{S}_2\text{O}_3$ decomposed by $\text{HCl}$ , with evolution of $\text{SO}_2$ and separation of $\text{S}$ .
As Group.	{	$\text{H}_3\text{AsO}_3$ precipitated by $\text{H}_2\text{S}$ as $\text{As}_2\text{S}_3$ (yellow). $\text{H}_3\text{AsO}_4$ "           "           "           " $\text{HIO}_3$ decomposed by $\text{HS}$ , with formation of an iodide and separation of $\text{S}$ .
Fe Group.	{	$\text{H}_2\text{CrO}_4$ precipitated by $(\text{NH}_4)_2\text{S}$ as $\text{Cr}_2(\text{HO})_6$ .

The following are additional tests for these acids:—

#### 10. SULPHUROUS ACID. $\text{H}_2\text{SO}_3$ , c.w. 82.

1.  $\text{BaCl}_2$  produces a white precipitate of  $\text{BaSO}_3$ , soluble in  $\text{HCl}$ . This solution, on addition of chlorine water, yields a white precipitate of  $\text{BaSO}_4$ , the Sulphite being oxidized to Sulphate.

2.  $\text{AgNO}_3$  produces a white precipitate of  $\text{Ag}_2\text{SO}_3$ , which darkens on heating, from precipitation of  $\text{Ag}$ .

\*3. Warmed with  $\text{HCl}$ ,  $\text{SO}_2$  is evolved with effervescence, and may be recognized by its smell, and by its turning green a solution of Potassium Bichromate, placed on a piece of filter paper, and exposed to the gas.

4.  $\text{H}_2\text{S}$  decomposes free  $\text{H}_2\text{SO}_3$  with separation of sulphur.

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11. THIOSULPHURIC (formerly called Hyposulphurous) ACID.  
 $\text{H}_2\text{S}_2\text{O}_3$ , c.w. 114.

1.  $\text{BaCl}_2$  produces a white precipitate of  $\text{BaS}_2\text{O}_3$ , soluble in  $\text{HCl}$ , with formation of sulphur as a yellow precipitate.

\*2.  $\text{HCl}$ , or  $\text{H}_2\text{SO}_4$  produces no immediate precipitate, but on standing a short time Sulphur is precipitated (yellow), and simultaneously  $\text{SO}_2$  is evolved.

3.  $\text{AgNO}_3$  produces a white precipitate of  $\text{Ag}_2\text{S}_2\text{O}_3$ , which rapidly darkens in colour and becomes ultimately black from formation of  $\text{Ag}_2\text{S}$ . These changes are hastened by heat.

4.  $\text{Fe}_2\text{Cl}_6$  produces a reddish colouration, but on heating it is decolorized, the Ferric being reduced to Ferrous Chloride.

12. ARSENIOS ACID.  $\text{H}_3\text{AsO}_3$ , c.w. 126.

\*1.  $\text{AgNO}_3$  produces in *neutral* solutions a yellow precipitate of  $\text{Ag}_3\text{AsO}_3$ , soluble in  $(\text{NH}_4)\text{HO}$ . If no precipitate appear at first owing to the solution not being neutral, add a few drops of a *very* dilute solution of  $(\text{NH}_4)\text{HO}$  until it appears.

2.  $\text{MgSO}_4 + \text{NH}_4\text{Cl} + (\text{NH}_4)\text{HO}$  produce no precipitate.  
 (See also reactions for Arsenic.)

13. ARSENIC ACID.  $\text{H}_3\text{AsO}_4$ , c.w. 142.

\*1.  $\text{AgNO}_3$  produces in neutral solutions a light-brown precipitate of  $\text{Ag}_3\text{AsO}_4$ . If necessary, add very dilute Ammonia, as in the preceding case.

2.  $\text{MgSO}_4 + \text{NH}_4\text{Cl} + (\text{NH}_4)\text{HO}$  produce a white precipitate of  $\text{MgNH}_4\text{AsO}_4$ . See also reactions for Arsenic.

14. IODIC ACID.  $\text{HIO}_3$ , c.w. 176.

1.  $\text{BaCl}_2$  produces a white precipitate of  $\text{Ba}(\text{IO}_3)_2$ , soluble in  $\text{HNO}_3$ .

2.  $\text{AgNO}_3$  produces a white crystalline precipitate of  $\text{AgIO}_3$ , readily soluble in  $(\text{NH}_4)\text{HO}$ , but sparingly soluble in  $\text{HNO}_3$ .

3.  $\text{SO}_2$  produces at first a precipitate of  $\text{I}$ , which is converted into  $\text{HI}$  on addition of excess of the re-agent.

\*4. On heating, iodates are decomposed, oxygen being evolved, and in some cases iodine is also given off in violet vapours.

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15. CHROMIC ACID.  $H_2CrO_4$ , c.w. 118.2.

1.  $BaCl_2$  produces a yellow precipitate of  $BaCrO_4$ , soluble in  $HCl$  and  $HNO_3$ , but insoluble in Acetic Acid.

2.  $H_2S$  in presence of  $HCl$  reduces the solution to  $Cr_2Cl_6$  (green), with separation of  $S$ . In neutral solutions,  $Cr_2(OH)_6$  is precipitated along with  $S$ .

3.  $SO_2$  reduces solutions of chromates to the chromic salt, the colour of which is green. Chromates are likewise reduced by zinc and a dilute acid, by Oxalic Acid and dilute Sulphuric Acid, by strong  $H_2SO_4$ , by strong  $HCl$ , and by boiling the solution acidified with  $HCl$  or  $H_2SO_4$  along with Alcohol.

4.  $AgNO_3$  produces a dark-red precipitate of  $Ag_2CrO_4$  soluble in  $HNO_3$  and in  $(NH_4)HO$ .

\*5. Lead acetate produces a bright yellow precipitate of  $PbCrO_4$ , soluble in  $NaHO$ , but soluble with difficulty in dilute  $HNO_3$ .

(See also reactions for Chromium.)

Reactions of the Acids belonging to Group III.

16. Acids precipitated by  $AgNO_3$ , and not by  $BaCl_2$ .

Hydrochloric, Hydrobromic, Hydriodic, Hydrocyanic, Hypochlorous, Nitrous, and Hydrosulphuric Acids.

HYDROCHLORIC ACID.  $HCl$ , c.w. 36.5.

1.  $AgNO_3$  produces a white curdy precipitate of  $AgCl$ , which becomes violet on exposure to light. The precipitate is insoluble in  $HNO_3$ , but soluble in  $(NH_4)HO$ , in  $KCN$ , in  $Na_2S_2O_3$ , and also to some extent in  $NaCl$ .

\*2. Heated with  $H_2SO_4$  and  $MnO_2$ , Chlorides yield chlorine gas, recognized by its smell, bleaching action and green colour.

3. *Dry* Chlorides, when heated in a retort with  $H_2SO_4$  and  $K_2Cr_2O_7$ , yield  $CrO_2Cl_2$  (chromium oxychloride), which distils over into the receiver as a dark red liquid, decomposed by addition of water or  $(NH_4)HO$ , yielding a yellow solution, which on addition of a lead salt, gives a yellow precipitate of  $PbCrO_4$ .

17. HYDROBROMIC ACID.  $HBr$ , c.w. 81.

1.  $AgNO_3$  produces a pale yellow precipitate of  $AgBr$ , insoluble in dilute  $HNO_3$ , soluble in strong  $(NH_4)HO$ , and readily in  $KCN$  and  $Na_2S_2O_3$ .

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\*2. Chlorine passed through a solution of a Bromide decomposes it with liberation of Br, which dissolves in the liquid and colours it yellow. If this solution be shaken up with Ether, the Bromine is dissolved by it, and the yellow ethereal solution floats above the liquid which becomes colourless. If the ethereal solution be then separated from the liquid, and NaHO be added, the yellow colour disappears and NaBr and NaBrO<sub>3</sub> are produced. On evaporation and ignition, Oxygen is evolved and NaBr alone remains, which may be tested as in 3.

3. Heated with H<sub>2</sub>SO<sub>4</sub> and MnO<sub>2</sub>, Bromides yield red vapours of Br, recognized by its powerful odour.

4. Heated in a retort with K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> and H<sub>2</sub>SO<sub>4</sub>, dry Bromides yield dark red vapours, which condense in the receiver to a liquid of the same colour which consists of pure Bromine, and is decolorized on adding excess of (NH<sub>4</sub>)HO. (Compare Hydrochloric Acid test).

#### 18. HYDRIODIC ACID. HI, c.w. 128.

1. AgNO<sub>3</sub> produces a pale yellow precipitate of AgI, insoluble in dilute HNO<sub>3</sub>, and very difficultly soluble in (NH<sub>4</sub>)HO, but readily in KCN and Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.

2. Cuprous sulphate\* produces a dirty-white precipitate of Cu<sub>2</sub>I<sub>2</sub>, which separates most completely if the solution be made slightly alkaline with Na<sub>2</sub>CO<sub>3</sub>. The reagent produces no precipitate in solutions of chlorides or bromides.

3. KNO<sub>2</sub> produces no reaction in solutions of Iodides until a few drops of HCl or H<sub>2</sub>SO<sub>4</sub> are added, when Iodine is at once liberated and colours the solution yellow. If a little starch solution be now added, a deep blue colouration results from the formation of Starch Iodide. On warming the blue liquid the colour disappears, but reappears on cooling. The production of Blue Starch Iodide is the most characteristic test for Iodine.

\*4. Chlorine water (or the gas) liberates Iodine from Iodides, but *excess* of Cl causes the formation of ICl<sub>3</sub>, which is colourless, and gives no blue colouration with starch solution. If therefore Chlorine Water be added drop by drop to a solution of an Iodide mixed with starch solution, a blue coloration is produced, which disappears on further addition of the reagent.

\*Prepared by dissolving a mixture of two parts CuSO<sub>4</sub> and five parts FeSO<sub>4</sub> in water, or by the action of SO<sub>2</sub> on CuSO<sub>4</sub>.

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5. Free Iodine (liberated by either of the above methods) is dissolved by  $\text{CS}_2$  forming a violet-coloured solution. If then, a solution of Iodine be shaken up with  $\text{CS}_2$ , the latter acquires a violet colour. Chloroform may be substituted for  $\text{CS}_2$ .

6. Heated with  $\text{MnO}_2$  and dilute  $\text{H}_2\text{SO}_4$ , violet vapours of Iodine are obtained, which colour paper moistened with starch, blue.

### 19.—HYDROCYANIC ACID. $\text{H}-\text{C}-\text{N}$ , c.w. 27.

1.  $\text{AgNO}_3$  produces a white precipitate of  $\text{AgCN}$ , insoluble in  $\text{HNO}_3$ , with difficulty in  $(\text{NH}_4)\text{HO}$ , but readily in  $\text{KCN}$  and  $\text{Na}_2\text{S}_2\text{O}_3$ .  $\text{AgCN}$  is decomposed on ignition, and metallic  $\text{Ag}$  remains; this serves to distinguish it from  $\text{AgCl}$ , which is not decomposed on ignition.

2. If a solution of  $\text{FeSO}_4$ , which has become oxidized by exposure to the air, be added to the solution of a Cyanide made alkaline with  $\text{NaHO}$ , a bluish green precipitate is formed, which is a mixture of Prussian blue with the hydrated oxides of iron. On adding  $\text{HCl}$ , these last are dissolved, and the blue precipitate remains.

\*3.  $\text{HCl}$  decomposes nearly all Cyanides with evolution of  $\text{HCN}$ , recognized by its odour, resembling bitter almonds. If a Cyanide be thus decomposed in a small porcelain basin, covered by a similar basin on which a drop of  $(\text{NH}_4)_2\text{S}_2$  (yellow) adheres, the latter is converted into  $(\text{NH}_4)\text{CNS}$ , which gives a blood-red colouration on addition of  $\text{Fe}_2\text{Cl}_6$  and  $\text{HCl}$ .

NOTE.— $\text{Hg}(\text{CN})_2$  cannot be detected by the above methods. The dry substance is detected by igniting in a small tube, when Cyanogen Gas is evolved, or the solution is decomposed by  $\text{H}_2\text{S}$  and filtered from the  $\text{HgS}$ ; the filtrate contains  $\text{HCN}$ .

### 20.—HYPOCHLOROUS ACID. $\text{HClO}$ , c.w. 52.5.

1.  $\text{AgNO}_3$  produces a white precipitate of  $\text{AgCl}$ .

2.  $\text{Pb}(\text{NO}_3)_2$  produces a white precipitate, which changes in colour to red, and ultimately to brown from formation of  $\text{PbO}_2$ .

3.  $\text{MnCl}_2$  produces a dark brown precipitate of  $\text{MnO}(\text{HO})_2$ .

4. Indigo and litmus solutions are decolourized, especially on addition of an acid.

\*5. Dilute acids decompose hypochlorites with evolution of  $\text{Cl}$ .  $\text{HNO}_3$  evolves  $\text{HClO}$  from hypochlorites.

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**21. NITROUS ACID.  $\text{HNO}_2$ , c.w. 47.**

1.  $\text{AgNO}_3$  produces a white precipitate of  $\text{AgNO}_2$ , soluble in a large excess of water.
2.  $\text{H}_2\text{S}$ , in presence of acid, produces a precipitate of S, and  $(\text{NH}_4)\text{NO}_3$  remains in solution.
- \*3.  $\text{FeSO}_4$ , in presence of an acid, produces a black colouration from solution of NO in the  $\text{FeSO}_4$ .

**22. HYDROSULPHURIC ACID (Sulphuretted Hydrogen).** $\text{H}_2\text{S}$ , c.w. 34.

1.  $\text{AgNO}_3$  produces a black precipitate of  $\text{Ag}_2\text{S}$ , insoluble in dilute acids.
2. Lead Acetate, even when highly dilute, produces a black precipitate of PbS.
3. Sodium Nitro-Prusside, in presence of NaHO, produces a reddish-violet colouration, even in very dilute solutions. The colour disappears in a very short time.
- \*4. HCl or  $\text{H}_2\text{SO}_4$  decomposes most sulphides with evolution of  $\text{H}_2\text{S}$ , recognized by its disagreeable odour, and by its blackening paper moistened with solution of lead.

**Reactions of the Acids belonging to Group IV.****23. Acids not precipitated by any reagent.****Nitric, Chloric, and Perchloric Acids.****NITRIC ACID.  $\text{HNO}_3$ , c.w. 63.**

1. Nitrates when heated evolve Oxygen, and in some cases nitrous vapours also. On fusing a nitrate and adding a fragment of charcoal, vivid deflagration occurs.

See Section I.

**24. CHLORIC ACID.  $\text{HClO}_3$ , c.w. 84.5.**

1.  $\text{H}_2\text{SO}_4$  decomposes chlorates with evolution of  $\text{Cl}_2\text{O}_4$ , a greenish yellow gas, having a powerful odour. If heated, violent explosions occur: the mixture ought therefore to be kept cold, and only *very* small quantities should be used.
2. When chlorates are heated, Oxygen is evolved and a metallic chloride remains, which may be dissolved in water and precipitated, as AgCl, by  $\text{AgNO}_3$ .

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3. Chlorates are reduced by  $\text{SO}_2$ , with liberation of Chlorine or its oxides, hence, if the solution of a chlorate be coloured blue with indigo solution, it is decolourized on adding  $\text{H}_2\text{SO}_4$  and solution of  $\text{Na}_2\text{SO}_3$ . (Distinction from perchlorates).

\*4.  $\text{HCl}$  decomposes chlorates with evolution of  $\text{Cl}$  and  $\text{Cl}_2\text{O}_4$ , a mixture called *euchlorine*.

5. Treated with charcoal, chlorates deflagrate violently.

#### PERCHLORIC ACID. $\text{HClO}_4$ , C.W. 100.5.

1.  $\text{H}_2\text{SO}_4$  does not act upon perchlorates in the cold, and on heating white fumes of  $\text{HClO}_4$  are given off, but no explosions occur.

\*2.  $\text{KCl}$  produce in strong solutions a white precipitate of  $\text{KClO}_4$ .

3. Indigo solution is not decolourized when added to perchlorates warmed with  $\text{HCl}$ , as *euchlorine* is not evolved.

4. Dry perchlorates evolve Oxygen on heating.

5. Anhydrous Perchloric Acid is a colourless fuming liquid, which explodes with great violence when dropped on wood charcoal. (*Roscoe*.)

6. Perchlorates are not reduced by  $\text{SO}_2$ .

#### 25.—TABLE G.

##### Detection of Inorganic Acids in Mixtures.

[The following acids are found in the examination for bases, which ought always to *precede* the examination for acids:— $\text{H}_2\text{SO}_4$ ,  $\text{H}_2\text{S}_2\text{O}_8$ ,  $\text{H}_2\text{CO}_3$ ,  $\text{H}_2\text{S}$ ,  $\text{Si}(\text{HO})_4$ ,  $\text{H}_2\text{CrO}_4$ ,  $\text{H}_3\text{AsO}_3$ , and  $\text{H}_3\text{AsO}_4$ .

##### (a) Acids in Soluble Bodies.

1. Neutralize a portion of the solution with  $(\text{NH}_4)\text{HO}$  and add  $\text{BaCl}_2$  (or  $\text{Ba}(\text{NO}_3)_2$  if  $\text{Ag}$ ,  $\text{Hg}_2$ , or  $\text{Pb}$  be present): precipitate indicates  $\text{H}_2\text{SO}_4$ ,  $\text{H}_3\text{PO}_4$ ,  $\text{H}_3\text{AsO}_3$ ,  $\text{H}_3\text{AsO}_4$ ,  $\text{Si}(\text{HO})_4$ ,  $\text{H}_2\text{CrO}_4$ ,  $\text{Ox}$ ,  $\text{Ci}$ ,  $\text{T}$ ,\* and large quantities of  $\text{B}(\text{HO})_3$  and  $\text{HF}$ .

To precipitate, add water and then  $\text{HCl}$ : if a precipitate remain,  $\text{H}_2\text{SO}_4$  was present.

2. To another portion of the neutralized solution add  $\text{AgNO}_3$ : a precipitate indicates one or more of the following acids:—

(a)  $\text{HCl}$ ,  $\text{HBr}$ ,  $\text{HI}$ ,  $\text{HCN}$ ,  $\text{H}_4\text{Fe}(\text{CN})_6$ ,  $\text{H}_3\text{Fe}(\text{CN})_6$ ,  $\text{H}_2\text{S}$ .

\*  $\text{Ox}$ ,  $\text{Ci}$ , and  $\text{T}$  are contractions for oxalic, citric and tartaric acids. (For the further separation of these organic acids, see Table H.)

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(b)  $H_3PO_4$ ,  $H_3AsO_4$ ,  $H_3AsO_3$ ,  $H_2CrO_4$ ,  $Si(HO)_4$ ,  $B(HO)_3$ ,  $\overline{Ox}$ ,  $\overline{T}$ , and  $\overline{Cl}$ . To the precipitate add cold dilute  $HNO_3$ . Acids under (a) are insoluble, those under (b) soluble.

DETECTION OF ACIDS UNDER (a).

To a portion of the solution add starch paste and one drop of a solution of  $N_2O_5$  in  $H_2SO_4$ . Blue colouration indicates **HI**. Add now chlorine water till the blue colour disappears, and shake with chloroform. If this becomes reddish-brown in colour, the presence of **H Br** is indicated. **HCl** is detected in the presence of the others by boiling down the solution to dryness and distilling the residue with  $K_2Cr_2O_7$  and  $H_2SO_4$ . See also note below.

DETECTION OF ACIDS UNDER (b).

Test separately for each acid by the methods already given.

SEPARATION OF  $H_3AsO_3$ ,  $H_3AsO_4$ , AND  $H_3PO_4$

Acidify solution with **HCl**, add  $Na_2SO_3$ , and heat until no smell of  $SO_2$  is given off. Pass  $H_2S$  through the hot solution, filter, and test for  $H_3PO_4$  with ammonium molybdate: yellow precipitate indicates  **$H_3PO_4$** . Precipitate another portion with magnesia mixture, and test both precipitate and filtrate for arsenic.

TEST FOR THE REMAINING ACIDS BY THE FOLLOWING REACTIONS GIVEN UNDER EACH ACID.

For **HCN** by test 3, 19.  
 For  $H_4Fe(CN)_6$  by tests 3 and 4, 32.  
 For  $H_3Fe(CN)_6$  by tests 2 and 3, 33.  
 For  $H_2S$ , by test 4, 22.  
 \*For  $HNO_3$ , see section 1.  
 For  $HClO_3$ , by tests 1 and 2, 24.  
 For  $\overline{Ox}$  and **HF**, by  $CaCl_2$ +acetic acid.  
 Confirm  $\overline{Ox}$  by test 4, 5, and **HF** by test 3, 6.

For  $B(HO)_3$  by tests 4 and 5, 4.  
 For  $Si(HO)_4$ , by tests 2 and 4, 8.  
 For  $H_2CrO_4$ , by tests 4 and 5, 15.  
 For  $H_2SO_3$  by test 3, 10, and smell of  $SO_2$  on adding **HCl**.  
 For  $CO_2$  by test 2, 7.  
 For  $H_2S_2O_3$  by tests 2 and 3, 11.

(b) Acids in Insoluble Bodies.

If the compound is not dissolved by the ordinary reagents, the substance must be fused with about four times its weight of a mixture of  $Na_2CO_3$  and  $K_2CO_3$ . When cold, extract the fused mass with water, and filter if necessary. The filtrate contains the acid, and is neutralized with **HCl** or  $HNO_3$ , and examined by the methods given under (a).

The sulphates of Barium, Strontium, and Calcium are decomposed by boiling with a concentrated solution of  $Na_2CO_3$ . Filter, and examine the filtrate for the acid.

Nitric Acid and Aqua Regia oxidize sulphides to sulphates: hence the solution of a sulphide in these acids always contains  $H_2SO_4$ . In such cases a separate portion of the substance must be examined for  $H_2SO_4$  by boiling with **HCl**, diluting with water, and then testing with  $BaCl_2$ .

NOTE.—In mixtures of chlorides, bromides, and iodides, or any two of them, proceed as follows:—Place a small quantity of the mixture in a test tube, add water and a few pieces of  $MnO_2$ † (free from chlorides), then one drop only of dilute Sulphuric Acid and boil: violet vapour indicates **Iodides**. Add another drop of the

\*If **HI** be present, it must first be removed by addition of  $FeSO_4 + CuSO_4$ .

†Powdered  $MnO_2$  produces too much "bumping" to be used for this purpose.

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dilute acid and boil again. Proceed in this way till no more violet vapour is given off. Then add about 2 c.c. dilute Sulphuric Acid and boil: brown vapour indicates **Bromides**. Boil till all bromine is expelled, allow to cool completely, add to the residue an equal bulk of strong Sulphuric Acid and warm; a green gas indicates **Chlorides**. Confirm by observing if a piece of moistened red blotting-paper held in the mouth of the tube is bleached.

**26.—Grouping of the Organic Acids.**

GROUP I. (TARTARIC ACID GROUP).

Group reagent,  $\text{CaCl}_2$ .

**Tartaric, Citric, and Malic Acids (Oxalic Acid, see 5).**

Acids which are precipitated by  $\text{CaCl}_2$  in the cold or on boiling.

GROUP II. (BENZOIC ACID GROUP).

Group reagent,  $\text{Fe}_2\text{Cl}_6$ .

**Benzoic and Succinic Acids.**

Acids which are not precipitated by  $\text{CaCl}_2$ , but which give precipitates with  $\text{Fe}_2\text{Cl}_6$  in neutral solutions.

GROUP III.

Group reagent,  $\text{AgNO}_3$ .

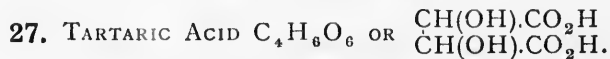
**Ferro-cyanic, Ferri-cyanic, Sulpho-cyanic, Acetic and Formic Acids.**

Acids precipitated by  $\text{AgNO}_3$  in neutral solutions, and not by  $\text{CaCl}_2$ , or  $\text{Fe}_2\text{Cl}_6$ . Acetates and Formates are only precipitated in concentrated solutions.

**Reactions of the Organic Acids belonging to Group I. (Tartaric Acid Group.)**

Acids precipitated by  $\text{CaCl}_2$  in the cold or on boiling.

**Tartaric and Citric Acids.**



1.  $\text{CaCl}_2$  in neutral solutions produces a white precipitate of  $\text{C}_4\text{H}_4\text{CaO}_6$ , soluble in acids, and in ammoniacal salts. The precipitate is soluble in  $\text{KHO}$ , but is re-precipitated when the solution is boiled, and on cooling is re-dissolved.

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\*2. KCL produces in solutions containing  $\bar{T}$  in excess a white crystalline precipitate of  $C_4H_5KO_6$ , soluble in mineral acids and alkalis, insoluble in Acetic Acid. The precipitation is promoted by stirring, or by addition of alcohol.

3. Lime-water produces in neutral solutions a white precipitate  $C_4H_4CaO_6$  (flocculent at first, afterwards crystalline), soluble in tartaric acid and  $NH_4Cl$ , but re-precipitated in crystals from these solutions after standing some time.

4. Add to some Calcium Tartrate which has been washed two or three times by decantation (after pouring off the wash water as completely as possible), a drop or two of  $(NH_4)HO$  and a crystal of  $AgNO_3$ , and heat the mixture in a test tube. A lustrous mirror of silver will deposit on the tube.

5. Heated with strong  $H_2SO_4$ , the mixture darkens rapidly from separation of Carbon, and  $SO_2$ ,  $CO$ , and  $CO_2$  are evolved.

6. Heated to redness, the substance darkens in color, and gives off the characteristic odour of burnt sugar.



1.  $CaCl_2$  produces no precipitate in neutral solutions in the cold, but on boiling,  $Ca_3(C_6H_5O_7)_2$  is precipitated, and is not soluble in  $KHO$ , but soluble in  $(NH_4)HO$ .

\*2. Lime-water produces no precipitate in cold neutral solutions, but on boiling,  $Ca_3(C_6H_5O_7)_2$  is precipitated.

3.  $AgNO_3$  produces in neutral solutions a white flocculent precipitate of  $C_6H_5Ag_3O_7$ , soluble in  $(NH_4)HO$ ; this solution does not blacken on boiling.

4. Heated with strong  $H_2SO_4$ ,  $CO_2$  and  $CO$  are evolved without any darkening in color; on continued heating, however, the mixture darkens, and  $SO_2$  is evolved.

5. Heated to redness, irritating fumes are given off, readily distinguished from those given off by heating the preceding acid.



1.  $CaCl_2$  produces no precipitate in neutral solutions in the cold, but upon boiling,  $C_4H_4CaO_5$  separates from strong solutions. The precipitate when heated with  $(NH_4)HO$  and  $AgNO_2$  causes no separation of silver.

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2. Lime water does not precipitate solutions of Malic Acid or of malates even on boiling. (Compare 28, 2.)

\*3.  $\text{AgNO}_3$  produces in neutral solutions a white granular precipitate of  $\text{C}_4\text{H}_4\text{Ag}_2\text{O}_5$ , which becomes grey on boiling.

4. Heated with strong  $\text{H}_2\text{SO}_4$ ,  $\text{CO}_2$  and  $\text{CO}$  are evolved, the fluid then darkens and  $\text{SO}_2$  is evolved.

Reactions of the Acids belonging to Group II. (Benzoic Acid Group.)

**30. Acids precipitated by  $\text{Fe}_2\text{Cl}_6$ , and not by  $\text{CaCl}_2$ .**

Benzoic and Succinic Acid.

BENZOIC ACID.  $\text{C}_7\text{H}_6\text{O}_2$ . ( $=\text{C}_6\text{H}_5$ .  $\text{CO}_2\text{H}$ ).

1.  $\text{Fe}_2\text{Cl}_6$  produces, in neutral solutions, a buff-colored precipitate of ferric benzoate, decomposed by  $(\text{NH}_4)\text{HO}$  with formation of a more basic Benzoate and Ammonium Benzoate. Ferric Benzoate is soluble in  $\text{HCl}$  with liberation of Benzoic Acid.

2. Heated with  $\text{H}_2\text{SO}_4$  Benzoic Acid does not blacken.

\*3. Heated in an open tube, the acid sublimes in needle-shaped crystals, and an irritating vapour is given off. When kindled, the crystals burn with a smoky flame.

**31. SUCCINIC ACID.**  $\text{C}_4\text{H}_6\text{O}_4$ . [ $=\text{C}_6\text{H}_4(\text{CO}_2\text{H})_2$ ]

1.  $\text{Fe}_2\text{Cl}_6$  produces, in neutral solutions, a reddish brown bulky precipitate of Ferric Succinate, soluble in mineral acids, and decomposed by  $(\text{NH}_4)\text{HO}$  in a similar manner to Ferric Benzoate.

2. Lead Acetate produces a white precipitate of Lead Succinate, soluble in excess of the reagent and in  $\text{HNO}_3$ .

\*3.  $\text{BaCl}_2$ , in presence of  $(\text{NH}_4)\text{HO}$  and alcohol, produces a white precipitate of Barium Succinate; this reaction serves to distinguish this acid from Benzoic, which does not give a similar precipitate.

4. Heated in an open tube, the acid sublimes in fine needles; and when kindled, the crystals burn with a bluish but not smoky flame.

Reactions of the Acids belonging to Group III.

**32. Acids precipitated by  $\text{AgNO}_3$ , in strong neutral solutions.**

Ferro-cyanic, Ferri-cyanic, Sulpho-cyanic, Acetic, and Formic Acids.

FERRO-CYANIC ACID.  $\text{H}_4\text{Fe}(\text{CN})_6$ . [ $=4\text{HCN}$ .  $\text{Fe}(\text{CN})_2$ ].

1.  $\text{AgNO}_3$  produces a white precipitate of  $\text{Ag}_4\text{Fe}(\text{CN})_6$ , insoluble in dilute  $\text{HNO}_3$  and in  $(\text{NH}_4)\text{HO}$ , but soluble in  $\text{KCN}$ .

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\*2.  $\text{CuSO}_4$  produces a reddish brown precipitate of  $\text{Cu}_2\text{Fe}(\text{CN})_6$ .

3.  $\text{Fe}_2\text{Cl}_6$  produces a deep blue precipitate of Prussian blue, insoluble in dilute mineral acids, soluble in Oxalic Acid, and decomposed by  $\text{NaHO}$  with separation of  $\text{Fe}_2(\text{HO})_6$ .

4.  $\text{FeSO}_4$  gives a light blue precipitate, which rapidly darkens in color by oxidation.

### 33. FERRI-CYANIC ACID. $\text{H}_3\text{Fe}(\text{CN})_6$ [= 3HCN. $\text{Fe}(\text{CN})_3$ ]

1.  $\text{AgNO}_3$  produces an orange-colored precipitate of  $\text{Ag}_3\text{Fe}(\text{CN})_6$ , insoluble in dilute  $\text{HNO}_3$ , soluble in  $(\text{NH}_4)\text{HO}$  and  $\text{KCN}$ .

\*2.  $\text{FeSO}_4$  produces a blue precipitate of  $\text{Fe}_3\text{Fe}_2(\text{CN})_{12}$  (Turnbull's blue, insoluble in acids, but decomposed by alkalies.)

3.  $\text{Fe}_2\text{Cl}_6$  produces no precipitate, but alters the colour to a greenish brown.

### 34. SULPHO-CYANIC ACID. HCNS. or H-S-C-N.

1.  $\text{AgNO}_3$  produces a white curdy precipitate of  $\text{Ag}(\text{CN})\text{S}$  insoluble in dilute acids, but soluble in  $(\text{NH}_4)\text{HO}$ , and in solution of  $\text{K}(\text{CN})\text{S}$ . From the solution in  $(\text{NH}_4)\text{HO}$  it crystallizes in shining scales.

2.  $\text{CuSO}_4$  produces in strong solutions a black crystalline precipitate of  $\text{Cu}(\text{CN})_2\text{S}_2$  which changes on standing to the cuprous salt  $\text{Cu}_2(\text{CN})_2\text{S}_2$  which is white. This change takes place at once by the addition of reducing agents (*e.g.*  $\text{SO}_2$  or  $\text{FeSO}_4$ ) to the cupric salt.

\*3.  $\text{Fe}_2\text{Cl}_6$  produces a blood-red colouration from formation of a soluble sulpho-cyanate of iron. The colour is destroyed by addition of alkalies, by  $\text{HgCl}_2$  and by many acids (*e.g.*  $\text{HNO}_3$ ,  $\text{H}_3\text{PO}_4$ ,  $\text{H}_2\text{C}_2\text{O}_4$ ,  $\text{HIO}_3$ ), but not by  $\text{HCl}$  even when concentrated.

### 35. ACETIC ACID. $\text{C}_2\text{H}_4\text{O}_2$ . (= $\text{CH}_3\cdot\text{CO}_2\text{H}$ .)

1.  $\text{AgNO}_3$  produces, in neutral solutions, a white crystalline precipitate of  $\text{C}_2\text{H}_3\text{AgO}_2$ , soluble in  $(\text{NH}_4)\text{HO}$  and in hot water.

2.  $\text{Fe}_2\text{Cl}_6$ , in neutral solutions, produces a deep red colouration. On boiling, all the iron separates as a light brown precipitate of basic acetate, and the fluid becomes colourless.

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3. \*A strong solution heated with  $H_2SO_4$  and Alcohol yields Acetic Ether, recognized by its characteristic and pleasant odour.

**36. FORMIC ACID.**  $CH_2O_2$ . (=  $H.CO_2H$ ).

1.  $AgNO_3$  produces, in neutral *concentrated* solutions, a white precipitate of  $CHAgO_2$ , which rapidly darkens, especially on heating, owing to separation of Ag.

2.  $Fe_2Cl_6$  produces a similar reaction with formates as with acetates.

\*3.  $Hg(NO_3)_2$  produces a white precipitate of the formate of mercury, which, however, soon becomes grey from separation of Hg.

\*4. *Cold strong*  $H_2SO_4$  decomposes formates with effervescence, CO being evolved. On heating, the gas comes off rapidly and if lighted, burns with a blue flame.

**37. STEARIC ACID.**  $C_{18}H_{36}O_2$ . (=  $C_{15}H_{33}.CO_2H$ ).

1. Heated with alkalis, a soap is formed. If mutton fat (which contains the acid combined with glycerine) be heated with NaHO, sodium stearate is formed, which dissolves in warm water and the glycerine separates. Soaps are decomposed by acids, which unite with the base, and the fat separates and may be dissolved in alcohol. From this solution the fat crystallizes in needles.

**38. SALICYLIC ACID.**  $C_6H_4$   $\begin{matrix} < \\ \text{OH} \\ \text{CO}_2\text{H} \end{matrix}$  (*o*)

1. Heated with lime phenol is evolved, easily recognized by its smell.

2. Ferric Chloride produces a deep violet colour, which disappears when treated with acids or alkalis.

3. Bromine Water gives a white precipitate.

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## 39. TABLE H.—SEPARATION OF ORGANIC ACIDS.

Remove the metals of the Silver, Copper, and Iron Groups by their respective group reagents. Neutralize the solution with  $(\text{NH}_4)_2\text{CO}_3$ , shake well, and allow to stand for at least twenty minutes.

PRECIPITATE.		FILTRATE.
<p><b>Oxalic and Tartaric Acids.</b></p> <p>Wash with water, warm precipitate with Acetic Acid, and filter.</p>		
RESIDUE.	FILTRATE.	
<p><b>CaOx.</b></p> <p>Confirm by tests 3 and 4, to ascertain the presence of Oxalic Acid.</p>	<p><b>CaT.</b></p> <p>Evaporate first to dryness and confirm by tests 4 and 5, to ascertain the presence of Tartaric Acid.</p>	
<p>Wash with Alcohol dissolve in HCl, add <math>(\text{NH}_4)_2\text{CO}_3</math>, boil and filter.</p>		
PRECIPITATE.	FILTRATE.	
<p><b>Citric, Malic and Succinic Acids.</b></p> <p>Wash with Alcohol, filter, wash with a little Alcohol, dissolve residue in <math>\text{HNO}_3</math> and evaporate to dryness on the water bath. Add excess of <math>\text{Na}_2\text{CO}_3</math> solution, boil, filter, neutralize exactly with HCl, heat and add solution of <math>\text{CaSO}_4</math> to a portion. A white precipitate indicates Malic Acid. To the remaining portion add CaCl<sub>2</sub> in excess, filter, and add Alcohol to the filtrate. A gelatinous precipitate indicates Succinic Acid.</p>	<p><b>Malic and Succinic Acids.</b></p> <p>Add Alcohol, filter, wash with a little Alcohol, dissolve residue in <math>\text{HNO}_3</math> and evaporate to dryness on the water bath. Add excess of <math>\text{Na}_2\text{CO}_3</math> solution, boil, filter, neutralize exactly with HCl, heat and add solution of <math>\text{CaSO}_4</math> to a portion. A white precipitate indicates Malic Acid. To the remaining portion add CaCl<sub>2</sub> in excess, filter, and add Alcohol to the filtrate. A gelatinous precipitate indicates Succinic Acid.</p>	
<p>Add to filtrate 3 volumes of strong Alcohol and filter.</p>		
PRECIPITATE.	FILTRATE.	
<p><b>Ferric Benzoate.</b></p> <p>Wash the precipitate, warm with <math>(\text{NH}_4)_2\text{CO}_3</math>, filter, evaporate filtrate nearly to dryness. Add HCl to obtain shining crystals of Benzoic Acid. Confirm by test 3, 30.</p>	<p><b>Benzoic Acid, Etc.</b></p> <p>Heat to expel Alcohol, neutralize exactly with HCl, and add <math>\text{Fe}_2\text{Cl}_6</math>. Filter.</p>	<p><b>Acetic and Formic Acid, Etc., Etc.</b></p> <p>(The filtrate is colored red in presence of Acetic or Formic Acid.) See Note.</p>
<p>Remaining Acids.</p>		

NOTE.—Acetic and Formic Acids are tested for in the original solution. Neutralize (if necessary) with  $\text{Na}_2\text{CO}_3$ , boil down to dryness, and test a portion for Acetic Acid by Ether test 35, 3. Or distil with  $\text{H}_2\text{SO}_4$  and digest distillate with excess of PbO to obtain Basic Acetate of lead. To another portion of dry residue add dilute  $\text{H}_2\text{SO}_4$  and distil. Heat part of the distillate with PbO to obtain crystals of lead formate. To another part add  $(\text{NH}_4)_2\text{CO}_3$  till neutral, and then  $\text{AgNO}_3$  (test r, 36), to ascertain the presence of Formic Acid.

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HO till neutral, and then  $\text{AgNO}_3$  (test I, 36), to ascertain  
the presence of **Formic Acid**.

## SECTION V.

### GOLD.

#### To obtain Pure Gold from a mixture of Metals.

Treat the mixture of metals in an evaporating dish with Aqua Regia (1 part  $\text{HNO}_3$  to 3 parts  $\text{HCl}$ ), keeping the mixture boiling. This should be done under a hood. This treatment should be continued, fresh acid having been added from time to time, until all the metals are dissolved and nothing remains but the white insoluble Silver Chloride. Filter.

#### RESIDUE.

Silver Chloride may be reduced on charcoal and a bead of metallic Silver obtained.

#### FILTRATE.

The filtrate should be boiled down nearly to dryness to expel excess of acid. Water is then added. To the clear liquid add a solution of  $\text{FeSO}_4$ , until the brown precipitate is no longer formed. Filter. Wash the precipitate with dilute  $\text{HCl}$ , with water, with  $\text{NH}_3$ - $(\text{OH})$ , and, lastly, again with water. The precipitated Gold may then be fused either in charcoal or in a crucible, a flux being used. The presence of other metals in the filtrate from Gold may be determined by means of the tables.

#### Quantitative determination of the amount of Gold present in an alloy.

Weigh out about one grain of the alloy to be examined, finely powdered. Treat this in a casserole with a solution of Aqua Regia. The treatment should continue, fresh acid being added from time to time, until all the alloy has been acted upon. Filter, being very careful that none of the liquid is lost in filtration. Evaporate the clear solution to the consistence of syrup, adding  $\text{HCl}$  from time to time. Dissolve the residue in water containing a drop of  $\text{HCl}$ . This solution is mixed with Oxalic Acid, or Ammonium Oxalate in excess, a little  $\text{H}_2\text{SO}_4$  being added. The vessel is covered with a glass plate, and is kept standing in a warm place for two days. At the end of this time the whole of the gold will be found to have separated in small yellow scales, which are collected on a filter,

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washed first with dilute HCl, then with water. The filter containing the gold is then dried. Place in a weighed-covered crucible and ignite. Weigh again; calculate the weight of the Gold and the percentage of it in the alloy.

#### PLATINUM.

Platinum is precipitated from a solution of its salts upon the addition of  $\text{FeSO}_4$  slowly and when boiled. If Gold and Platinum are present it is better to precipitate the gold present by means of Oxalic Acid. This reagent does not precipitate platinum. The Gold may then be filtered from the solution and the platinum precipitated by means of  $\text{FeSO}_4$ , or by adding some metallic Zinc to the solution, in which case the precipitation is much quicker.

#### SILVER.

##### To obtain Pure Silver.

Treat commercial or coin Silver with Nitric Acid (50%) in an evaporating dish. Heat gently on a sand bath. When the whole has been dissolved add an equal bulk of distilled water. Filter the solution if necessary. Why might it be necessary? To the clear liquid add NaCl until no further precipitation takes place. Filter. Wash the precipitate thoroughly until the wash water shows no blue colouration upon the addition of  $\text{NH}_4(\text{OH})$ . What would a blue colouration indicate? The Chloride should now be transferred to a Beaker, and twice its bulk of water, containing 10 per cent. of  $\text{H}_2\text{SO}_4$ , added. Several bath nails are now added, the whole being kept stirred. What reaction takes place? When the colour of the mass changes from white to dark grey the reaction is complete. The pieces of iron are now removed, and the precipitated Silver is washed first with dilute HCl, and then with distilled water, repeating the operation several times. Finally the Silver is dried.

#### MERCURY.

Commercial Mercury is often impure. Such impurities as cannot be removed by forcing through chamois skin are probably Lead, Tin, Zinc and Bismuth. Impure Mercury has often a dull appearance, leaves a trace on white paper, does not form perfect globules, and coats the inside of a bottle with a dark coating. If treated with strong  $\text{H}_2\text{SO}_4$  many of the impurities become oxidized and dissolve. The best way to purify Mercury, however, is by distillation.

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Shake up in a test tube some Mercury, with equal parts Acetic Acid and water. To the liquid add a solution of Potassium Iodide. A yellow precipitate indicates Lead.

Dissolve a little Mercury in Nitric Acid without heat. If on the addition of a quantity of water a white precipitate is formed, Bismuth is present. Write the formula for the reaction.

If Mercury containing Tin is dissolved in  $\text{HNO}_3$ , a white, flaky residue of Tin Oxide remains.

Boil Mercury in  $\text{HCl}$ , decant the liquid and add  $\text{H}_2\text{S}$ . If no precipitate forms the Mercury was pure.

### TIN.

#### To obtain chemically Pure Tin.

Ordinary commercial Tin is often contaminated with Arsenic, Copper, Iron, Antimony, etc. To obtain chemically pure Tin dissolve commercial Tin in  $\text{HCl}$ . Write the equation. What becomes of the Arsenic and Antimony present? How would you determine their presence? The liquid is now evaporated to a small bulk. Nitric Acid is now added, when white, crystalline, insoluble, metastannic acid is formed. The whole is evaporated to dryness. Add water acidulated with  $\text{HCl}$ . Filter; wash several times; dry. Melt the residue with charcoal in a crucible. Chemically pure Tin will be the result.

#### EXAMINATION OF AMALAGAMS AND ALLOYS.

The substance to be examined is heated in a hard glass tube closed at one end. If Mercury is present it will volatilize, and part of it will collect in the upper part of the tube, forming a mirror. Heat until all the Mercury has been given off.

The substance should now be powdered as thoroughly as possible, transferred to a flask or beaker, and enough  $\text{HNO}_3$  added to dissolve it with the aid of gentle heat. The appearance of the solution will often afford a clue as to its constituents. If the liquid be coloured blue, Copper is present. If the precipitated metastannic acid is white, Gold and Platinum are present in no considerable quantities. If, however, the metastannic acid is coloured purple, Gold is present. The presence of a black powder with the metastannic acid indicates the presence of Platinum in small particles.

The contents of the flask should be filtered. The presence of the white metastannic acid indicates Tin.

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If the presence of Antimony is suspected, the metastannic acid should be dehydrated, forming  $\text{SnO}_2$ , by heating into a red heat for some time. Place this in a silver crucible, add some  $\text{NaOH}$  and heat for some time.  $\text{NaSbO}_3$  and  $\text{NaSnO}_3$  will be formed. The fused mass is now powdered and treated with cold water and filtered. If Antimony be present it will appear as insoluble Antimonate of Sodium.

To some of the original solution add  $\text{HCl}$  until no further precipitation takes place. The precipitate may contain Silver or Lead. Test for these by table A, Section III.

If Copper or Cadmium are present they may be precipitated from the original solution by the addition of  $\text{H}_2\text{S}$ . The sulphide of Copper will be black, that of Cadmium being yellow. The method of separation to be followed is that of table B, Section III.

To the original solution add  $(\text{NH}_4)_2\text{CO}_3$ . A white precipitate indicates the presence of  $\text{ZnCO}_3$ .

Treat a small quantity of the original substance, powdered with aqua regia. Heat; boil down to dryness; add water; filter. To the clear liquid add Oxalic Acid and Ammonium Oxalate. If Gold be present it will be precipitated. Filter. To the clear solution add  $\text{FeSO}_4$ , or metallic Zinc, when Platinum, if present, will be precipitated slowly, and on boiling in the first case, more quickly in the second.

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#### TESTS FOR CEMENTS.

##### 1. OXYPHOSPHATE CEMENTS.

1. Pour a little liquid into a test tube. Hold the latter inclined, and down the side pour an aqueous solution of egg albumen. A white band at the junction indicates Metaphosphoric Acid.

2. To a solution of Silver Nitrate add a little glacial Phosphoric Acid. A white precipitate is formed.

3. To some of a solution add Barium Chloride. A white precipitate is formed.

The liquid should always answer to all three of these tests.

##### 2. OXYCHLORIDE OF ZINC CEMENTS.

1. To show that the liquid contains Zinc.

(a) When  $\text{HCl}$  is added no precipitate should be formed.

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(b) Pass  $H_2S$  through some of the solution. No precipitate should be formed.

(c) To a fresh amount add  $NH_4(OH)$ ,  $NH_4Cl$  and  $(NH_4)_2S$ . A white precipitate should be formed.

(The precipitate will be green in the presence of iron.)

Place some of the liquid in a test tube. Cautiously allow some  $NH_4(OH)$  to trickle down the side of the tube. A faint band of turbidity should be noticed.

2. To prove that it is a Chloride add some of it to a solution of  $AgNO_3$ . A white curdy precipitate, becoming dark when allowed to stand in the light, indicates the presence of a Chloride.

The powder should be tested for Zinc in the dry way and in the wet way. The usual tests for (1) Chlorides, (2) Sulphates (3) for Lead, and (4) for Iron, should give no reactions if the substance is pure.

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## SECTION VI.

### EXPERIMENTS WITH CERTAIN ORGANIC COMPOUNDS.

#### I. ALCOHOLS. ETHYL ALCOHOL. $C_2H_5(OH)$ .

The chief impurities present in Ethyl Alcohol may be water and fusel oil. The latter, which consists chiefly of Amylic Alcohol, may be removed by distillation. The former may be removed by distillation from quicklime.

Determine the properties of pure Alcohol, including colour, odour, taste, boiling point, inflammability. Does its vapour mixed with air explode? Does it solidify when cold? Is it a good solvent?

#### METHYL ALCOHOL. $C_2H_3(OH)$ .

Make a list of the properties of Methyl Alcohol. What are Methylated Spirits?

#### GLYCEROL (GLYCERINE) $C_3H_8O_3 = [C_3H_5(OH)_3]$

Note the thick, syrupy consistence, sweet taste, solubility in water and alcohol and absence of inflammability.

Warm a solution of glycerol in water with a little  $H_2SO_4$  and note the odour of acrolein.

Find out whether it will reduce an alkaline copper solution.

#### PHENOL. (CARBOLIC ACID). $C_6H_5(OH)$

Note the colour and odour of the crystals; also, their effect upon the skin.

Bromine water gives a yellowish-white precipitate (Tribromophenol) even in dilute solutions.

Ferric Chloride produces a violent colouration.

Heated with Ammonia and a drop of Sodium Hypochlorite, a deep blue colouration is produced which becomes red on addition of acids.

To a few crystals in a test tube, add a little Nitric Acid. What is formed?

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II. CHLOROFORM.  $\text{CHCl}_3$ .

Note its odour, taste, solubility in water and alcohol.

Drop a piece of filter paper on it. Light this. What is the product of combustion? Prove it.

Pure Chloroform should not affect Litmus, nor colour green a mixture of  $\text{H}_2\text{SO}_4$  and  $\text{H}_2\text{CrO}_4$ .  $\text{H}_2\text{SO}_4$  nor  $\text{K}(\text{OH})$  should not colour it brown. Allowed to evaporate on a sheet of white paper no residue nor colour should remain.

To purify Chloroform, agitate it first with  $\text{H}_2\text{SO}_4$ . Draw off the acid, then treat with a solution of  $\text{Na}_2\text{CO}_3$ . Separate the two. Then wash with water and distil over Quicklime.

How is Chloroform prepared?

III. ETHER.  $(\text{C}_2\text{H}_5)_2\text{O}$ .

Note the properties of Ether.

Pure Ether should not affect blue Litmus, should leave no residue when evaporated, nor impart a blue colour to ignited Copper Sulphate.

Ether may be purified by washing with dilute Caustic Soda solution, then with distilled water. It should then be distilled with half its weight of Calcic Chloride, taking care that the temperature does not rise above  $50^\circ\text{C}$ ., and that no flames come in contact with it. The distillate should be collected in a vessel surrounded by ice water.

How is Ether made?

## IV. CARBO-HYDRATES.

GRAPE SUGAR  $\text{C}_6\text{H}_{12}\text{O}_6$ .

$\text{H}_2\text{SO}_4$  forms with Grape Sugar a definite chemical compound of a yellow colour. No charring takes place.

Grape Sugar reduces an alkaline copper solution. Fehling's solution is the one usually used. It is prepared by dissolving 34.64 grains of pure Copper Sulphate in water, adding a solution of 200 grains Potassium Sodium Tartrate (Rochelle Salt), and 600 to 700 grains Caustic Soda of a specific gravity 1.12, and diluting so that the whole makes one litre. In a test tube boil about 10 c.c. of the above solution. To it add a few drops of a dilute solution of Glucose. A red precipitate is formed. What is it? Begin by using as little as possible of the Glucose solution, and endeavor to make the test as delicate as possible.

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CANE SUGAR  $C_{12}H_{22}O_{11}$ .

Treat a solution of Cane Sugar with strong  $H_2SO_4$ .

Does Cane Sugar reduce Fehling's solution. Try it? Add a drop of  $H_2SO_4$  to the Cane Sugar solution; then test with Fehling's solution. What is the action of the  $H_2SO_4$ ?

STARCH.  $(C_6H_{10}O_5)_x$ .

See the test for Iodine.

## V. ALBUMIN.

1. Boiling water causes Albumin to coagulate and it becomes at the same time insoluble, so that if a solution in cold water be heated, coagulation at once takes place.

2.  $HgCl_2$  coagulates Albumin, even when present in exceedingly small quantities. Coagulation also takes place with solutions of other salts, *e.g.*  $CuSO_4$ ,  $SnCl_2$ ,  $AgNO_3$ .

3. Most acids precipitate Albumin from its solutions.  $HNO_3$  acts most readily, and is therefore used to detect the presence of dissolved Albumin. Acetic, Tartaric, and Ortho-phosphoric Acids do not coagulate Albumin, except in very concentrated solutions.

## VI. CASEIN—MILK AND CHEESE.

1. Casein is insoluble in water, but is kept in solution in milk by a small quantity of free alkali. Such a solution does not coagulate by heat, but a film forms on its surface when heated. If this film be removed another forms in its place.

2. All acids (except Carbonic) precipitate Casein from its solutions; the precipitate is soluble in excess of the reagent.

3. Infusion of rennet (the inner membrane of the stomach of a calf) coagulates Casein completely.

VII. UREA.  $CH_4N_2O$ .

1.  $HNO_2$ , when added to solutions of Urea, unites with it, forming the nitrate, which separates out in crystalline plates, which are tolerably soluble in hot water and alcohol, but nearly insoluble in  $HNO_3$ .

2.  $KHO$  added to Urea decomposes it on heating, and  $NH_3$  is evolved.

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3.  $\text{Hg}(\text{NO}_3)_2$  produces a white precipitate of variable composition.

4.  $\text{HNO}_2$  decomposes Urea at once into  $\text{CO}_2$ ,  $\text{H}_2\text{O}$ , and  $\text{N}$ .  
 $\text{CH}_4\text{N}_2\text{O} + 2 \text{HNO}_2 = \text{CO}_2 + 4\text{N} + 3\text{H}_2\text{O}$ .

5. When Chlorine is passed into an aqueous solution of Urea the following reaction takes place:  $\text{CH}_4\text{N}_2\text{O} + 6 \text{Cl} + \text{H}_2\text{O} = \text{CO}_2 + 2\text{N} + 6\text{HCl}$ .

#### VIII. THE ALKALOIDS.

##### ACONITINE. $\text{C}_{33}\text{H}_{43}\text{NO}_{12}$ (ACONITE\*)

1. The solution in concentrated  $\text{H}_2\text{SO}_4$  is yellowish-brown, which becomes bright yellow upon the addition of dilute  $\text{HNO}_3$ .

2. The solution in aqueous phosphoric acid shows a yellow colour when evaporated.

##### ATROPINE. $\text{C}_{17}\text{H}_{23}\text{NO}_3$ (ATROPA BELLADONNA).

1. The solution in  $\text{HNO}_3$  is colourless, and on addition of  $\text{K}_2\text{Cr}_2\text{O}_7$  is coloured very slowly.

2. Add a little  $\text{HNO}_3$  to the dry substance, dry on the water bath, cool, and add a few drops of an alcoholic solution of  $\text{KHO}$ ; a violet colour, changing slowly to red, will be noticed.

3. Dissolve a fragment of  $\text{K}_2\text{Cr}_2\text{O}_7$  in  $\text{H}_2\text{SO}_4$ , add a grain of Atropine and a few drops of water, warm, when an odour of orange blossom will be perceived.

##### BRUCINE. $\text{C}_{23}\text{H}_{26}\text{N}_2\text{O}_4 \cdot 4\text{H}_2\text{O}$ (Strychnos nux vomica and St. Ignatius Bean).

1.  $\text{HNO}_3$  dissolves Brucine, producing an intense red colouration, which becomes yellow on heating. If  $\text{SuCl}_2$ ,  $\text{Na}_2\text{S}_2\text{O}_3$  or  $(\text{NH}_4)_2\text{S}$  be added, the colour changes to violet.

##### CINCHONINE. $\text{C}_{20}\text{H}_{24}\text{N}_2\text{O}$ (Cinchona Bark).

1. Chlorine water produces no colouration, even on the addition of  $(\text{NH}_4)\text{HO}$ , which produces a yellowish precipitate.

2.  $\text{KHO}$  or  $(\text{NH}_4)\text{HO}$  produces, in solutions of Cinchonine Salts, a white amorphous precipitate of Cinchonine, insoluble in excess, and not dissolved when shaken up with Ether. (Compare test 2 for Quinine. 68.)

\*The names in parenthesis indicate the source from which the alkaloid is derived.

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3. Solutions of Cinchonine have a bitter taste, and an alkaline reaction. The salts are less soluble in water and alcohol than those of Quinine.

COCAINE.  $C_{17}H_{21}NO_4$  (Cocoa Leaves.)

1. The solutions in  $H_2SO_4$  and  $HNO_3$  are colourless.
2.  $NH_4OH$  produces a white precipitate, soluble in excess.

CONINE.  $C_8H_{17}N$ . (Conium Masculatum.)

1. Potassium Mercuric Iodide produces a resinous precipitate, which becomes crystalline on standing.
2. Chlorine water produces a turbidity.

CODEINE.  $C_{18}H_{21}NO_3 \cdot H_2O$ . (Opium.)

1. Solution in  $HNO_3$  is yellow.
2. Solution in Chlorine water is colourless, but reddened by Ammonia water.
3. Solution in pure  $H_2SO_4$  is colourless, but turns blue when warmed with the addition of a trace of Ferric Chloride.

CAFFEINE OR THEINE.  $C_8H_{10}N_4O_2 \cdot H_2O$ . (Tea, Coffee).

1. The solution in  $HNO_3$  is yellow. Evaporate and warm with  $NH_4(OH)$ , when it turns purple.
2. If Chlorine or Bromine water is added to a solution, and the mixture evaporated to dryness, and the reddish-brown mass moistened with  $NH_4OH$ , a violet colouration will be produced.

MORPHINE.  $C_{17}H_{19}NO_3$ . (Opium).

1. KHO and  $(NH_4)HO$  precipitate Morphia from its salts, readily soluble in excess of KHO, less readily in excess of  $(NH_4)HO$ .
2. Concentrated  $HNO_3$ , added to powdered Morphia or its salts, produces an orange red colouration, changing afterwards to yellow. The reaction is best seen on a porcelain crucible lid. Addition of  $SnCl_2$  or of  $Na_2S_2O_3$  destroys the colour.
3. A neutral solution of  $Fe_2Cl_6$  produces, when added to Morphia (either in the dry state or in solution), a deep blue colour.
4.  $HIO_3$ , when added either to free or combined Morphia, is decomposed with liberation of I, which colours the liquid brown. Starched paper added to the solution becomes blue. Addition of  $(NH_4)HO$  deepens the brown colour.

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5.  $H_2SO_4$  produces no colouration with Morphia or its salts, but on adding a crystal of  $K_2Cr_2O_7$  a bright green colour is produced.

NARCEINE.  $C_{23}H_{29}NO_9 \cdot 2H_2O$ . (Opium).

1. A weak, Iodine solution, colours solid Narceine dark blue.
2. Concentrated  $H_2SO_4$  turns it brown, but when added in excess produces a bright yellow colour.

NARCOTINE.  $C_{22}H_{23}NO_7$ . (Opium.)

1. Concentrated  $H_2SO_4$  containing  $HNO_3$  produces an orange yellow colouration, which changes to red and finally to violet.
2. Bromine water produces a yellowish green colouration, which changes to a yellowish red upon the addition of Ammonia.

NICOTINE.  $C_{10}H_{14}N_2$ . (Tobacco.)

1. Concentrated  $HNO_3$  produces a red colouration.
2. Potassium Mercuric Iodide produces a resinous precipitate, becoming crystalline on standing.

QUININE.  $C_{20}H_{24}N_2O_2 \cdot 3H_2O$ . (Cinchona Bark.)

1. Chlorine water, when added to an acid solution of Quinine or its salts, produces no colouration until  $(NH_4)HO$  is added, when a bright green colour is produced. If  $K_4Fe(CN)_6$  be added before the  $(NH_4)HO$ , a red colouration is produced, soon changing to dirty brown.
2.  $KHO$  or  $(NH_4)HO$  produces, in solutions of Quinine salts, a white amorphous precipitate of Quinine, which, on standing, becomes crystalline. The precipitate is scarcely soluble in  $KHO$ , but slightly soluble in  $(NH_4)HO$ ; if a mixture of a Quinine solution and  $(NH_4)HO$  be shaken up with Ether, the precipitated Quinine is dissolved.
3. Solutions of Quinine or its salts are fluorescent in a high degree, and possess an extremely bitter taste.

STRYCHNINE.  $C_{21}H_{22}N_2O_2$  (Nux Vomica).

1. Strong  $H_2SO_4$  dissolves Strychnine without any colouration, even when heated to  $100^\circ C$ . Certain characteristic colours are produced :—
  - (a)  $PbO_2$ , blue colouration, changing to violet, then red, and finally yellow.

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(b)  $K_2Cr_2O_7$ , blue colouration, changing soon to yellowish-red.

(c)  $K_4Fe(CN)_6$ , violet colouration, changing less quickly.

(d)  $MnO_2$ , violet colouration, changing to dark-red.

2 Strong  $HNO_3$  dissolves Strychnine without colouration unless heated, when it becomes yellow.

3. The taste of Strychnine is intensely bitter, and is perceptible even in the most dilute solutions.

VERATRINE.  $C_{32}H_{49}NO_9$  (Seeds of *Veratrum Sabadilla*.)

1. The solution in  $H_2SO_4$  is first yellow, then orange, finally carmine-red, and shows a partial green fluorescence.

2. The solution in  $HCl$  is colourless, but turns dark-red when warmed.

3. Addition of Bromine water colours the substance violet.

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## SECTION VII.

### I. THE TESTING OF WATER.

#### (a) For Organic Impurities.

1. Place the water to be tested in a Florence flask, and add to it, first, a few drops of Sulphuric Acid, and then enough of a solution of Permanganate of Potash to give to the whole a deep purple tint. Set to one side for an hour or two, in a warm place, and if the solution loses its colour, organic impurities are present.

2. Fill a bottle with the water to be tested and cork it very tightly. Set aside in a warm place for a few days and then examine. An offensive odour indicates the presence of much organic matter.

#### (b) Temporary Hardness.

1. Half fill a large test tube with clear Lime-water, and then pass into it a stream of Carbon Dioxide. At first the usual result follows; but as the stream is continued the milkiness gradually disappears on account of the formation of a supposed Bicarbonate of Lime  $H_2Ca(CO_3)_2$ , which, unlike the Carbonate, is soluble in water. Water with the bicarbonates of Calcium or Magnesium and of iron dissolved in it, is said to be temporarily hard.

2. Divide the water obtained in the preceding experiment into three parts, and perform the following operations:

(a) To the first part add some of Clark's\* soap solution.

(b) Boil the second part of the solution for half an hour. Then filter and test the filtrate with Clark's solution.

(c) Add carefully, to the third part, some clean Lime-water. Filter, and test with Clark's solution.

#### (c) Permanent Hardness.

1. Fill a large test tube or small Florence flask about three-fourths full of pure water. Then add a gram or two of calcic sulphate  $CaSO_3$ , and shake vigorously. Filter. The clear water contains calcic sulphate in solution and is *permanently* hard.

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\*Clark's Solution consists of a solution of 10 grains of Castile Soap in 1 litre of 35 per cent. Alcohol.

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2. Divide the water thus obtained into four parts, and perform the same experiments with three parts of it that you did with the water which had Bicarbonate of Calcium dissolved in it.

3. To the fourth part add a solution of washing soda,  $\text{Na}_2\text{CO}_3$ . Note the result carefully.

Filter and test the filtrate as in the preceding cases with Clark's solution.

## II. QUALITATIVE TESTS FOR THE NORMAL CONSTITUENTS OF SALIVA.

1. Boil a little Saliva in a long slender test tube, held by the closed end between the thumb and finger. Heat the upper part only. A turbidity indicates the presence of Albumin.

2. To a fresh supply of Saliva add a drop or two of Ferric Chloride. A blood red colouration indicates the presence of Sulpho Cyanide.

This test may be performed by preparing a test paper made by dipping a piece of filter paper in an acid solution of Ferric Chloride. A drop of Saliva placed on one of these test papers should leave a red spot.

3. Collect a plentiful supply of Saliva, add four times its volume of water, stir well, let settle and pour off the clear liquid from the sediment. Prepare some starch mucilage by rubbing a little starch in cold water, then pouring into 500 cc. hot water, and then boil until the liquid is clear. Pour some of the starch into a small beaker. To this add some of the diluted Saliva and keep at a temperature of  $95^\circ$  to  $104^\circ$  for ten minutes. At the end of ten minutes test for starch and sugar by the Iodine and Fehling's tests respectively. What change has taken place? What has caused this change?

4. Fill a test tube with dilute Acetic Acid (one of acid to three of water.) Let Saliva drop slowly into it. Stringy flakes indicate the presence of Mucin.

5. To show the presence of inorganic acids evaporate a quantity of Saliva until the organic matters are thoroughly charred. When cool, add distilled water, stirring well and adding a drop or two of Acetic Acid. Filter and divide the filtrate into five parts.

(a) To one add a drop of  $\text{AgNO}_3$ . A turbidity indicates the presence of Chlorides.

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(b) To a second add a drop of  $\text{HNO}_3$  and then some Ammonium Molybdate solution. A yellowish colour indicates the presence of Phosphates.

(c) To a third add a drop or two of  $\text{HCl}$  and then some  $\text{BaCl}_2$  solution. A white precipitate shows the presence of Sulphates.

6. (a) To another portion add some Ammonium Oxalate solution. A white precipitate indicates the presence of Calcium.

(b) To a second part add Ammonia and Sodium Phosphate solution. A white precipitate indicates the presence of Magnesium.

### III. ANALYSIS OF TEETH.

1. Digest the teeth for a day or two in dilute  $\text{HCl}$  (10 per cent.) The earthy matter will be dissolved out, leaving only the soft and elastic organic matter.

2. To show the presence of earthy salts, place a few teeth over a Bunsen flame and heat strongly until the organic matter is all driven off. Powder and dissolve in  $\text{HCl}$ . Dilute and add plenty of  $\text{NH}_4(\text{OH})$ . A white gelatinous precipitate indicates the presence of the *Phosphates of Lime and Magnesia*. Filter, and to the filtrate add Ammonium Oxalate. A white precipitate indicates Calcium, not as a phosphate. The presence of carbonates may be proved by digesting teeth in Hydrochloric Acid and testing the gas which comes off with lime water. What gas is it?

### IV. TESTS FOR THE ABNORMAL CONSTITUENTS OF URINE.

1. Obtain perfectly clear Urine by filtering. Place the clear Urine to the depth of an inch in a test tube. Allow colourless Nitric Acid to flow down the side of the incline of the test tube into the Urine. A clear cut white band of coagulated Albumin at the juncture of the Urine and the acid will show itself if present. Confirm by taking a fresh quantity of Urine, filling a test tube two-thirds full, and adding a drop or two of Acetic Acid. Heat the upper end of the tube. A turbidity in the heated portion indicates Albumin.

2. Remove the Albumin, if present, from some Urine by heating with a drop or two of Acetic Acid and filtering. Test for sugar, using Fehling's solution.

3. Place a little Urine on a white plate—a crucible cover will do—add  $\text{HNO}_3$ . A peculiar play of green, yellow and violet colours occurs if bile is present. Agitate concentrated Urine with boiling Ether. The solution is greenish yellow if bile is present.

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## DETERMINATION OF ALBUMEN IN URINE.

Measure the Urine passed in 24 hours. Drop 50 cc., one cc. at a time, into one ounce boiling distilled water in a porcelain dish. If the Urine was alkaline, add a drop of Acetic Acid, but no more. Allow the coagulated Albumen to settle. Filter through a weighed crucible and wash well. Dry at  $100^{\circ}$  C. and weigh. Calculate the percentage of Albumen present in the whole amount.

## DETERMINATION OF SUGAR IN URINE.

Prepare Fehling's solution as follows:—Dissolve exactly 34.639 grams of dry  $\text{CuSO}_4$  in about 200 cc. of water. In another vessel dissolve 173 grams C. P. Rochelle salts  $\text{KNaC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$  in 480 cc. pure Na (OH) solution, having a specific gravity of 1.14. Mix the solutions and dilute to exactly 1000 cc. 10 cc. of this solution contains .34639 grams  $\text{CuSO}_4$ , and corresponds to 0.050 grams Anhydrous Glucose. Dilute the Urine 5 or 10 times.

Run exactly 10 cc. of Fehling's solution into a small flask. Add 40 cc. of water, heat to boiling, and run into the solution very gradually from an accurate burette some of the Urine. Continue until the last shade of bluish green disappears and a small portion of the liquid filtered gives no reaction with  $\text{H}_2\text{S}$ , nor with  $\text{HC}_2\text{H}_3\text{O}_2$  and  $\text{K}_4\text{FeCN}_6$ .

Since 10 cc Fehling's solution corresponds to 0.050 grams Anhydrous Glucose, the reading shows the amount of Urine containing 0.050 grams Glucose from which the percentage may be determined.

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