Qualitative chemical analysis / by C. Remigius Fresenius.

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QUALITATIVE CHEMICAL ANALYSIS.

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BY

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Seventh Edition.

EDITED BY

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PREFACE.

THIS edition differs greatly from the last. Several improvements, necessitated by the progress of discovery, have been introduced by the author; and I have specially striven to meet the present wants of English students. The language has been condensed, the notation and nomenclature have been modernized, the arrangement has been simplified. The consideration of rare inorganic bodies and of organic bodies has been deferred to the latter part of the volume where a section has been devoted to each. The grouping of the metals has been harmonized with the course of analysis. The course of analysis has been simplified in description, and the preliminary examination has been curtailed. Analytical tables have been added at the end, which include the preliminary examination and the detection of metals in soluble mixtures. The tables constitute merely a skeleton of the method given in the text; they are intended for the use of students who have made themselves thoroughly acquainted with the latter.

All the facts and, except in one or two trivial instances, all the processes given in this edition have the authority of Fresenius, but the principal changes in the arrangement have been made on my own responsibility.

A. V.

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REAGENTS, APPARATUS,

AND

MANIPULATION.

REAGENTS.

LIST OF REAGENTS.

Dry Reagents.

Borax	$Na_2B_2O_4 . B_2O_3$	In powder.
Microcosmic salt	$NaNH_4HPO_4$	See p. 5.
Potassium and sodium carbonate	$KNaCO_3$	See p. 5.
Potassium chlorate	$KClO_3$	
Potassium cyanide	KCN	See p. 6.
Potassium nitrate	KNO_3	In powder.
Potassium acid sulphate	$KHSO_4$	See p. 6.
Sodium carbonate	Na_2CO_3	Commercial, anhydrous.

Wet Reagents.

Alcohol		Methylated spirit. See p. 6.
Ammonium carbonate	$(NH_4)_2CO_3$	See p. 6.
Ammonium chloride	NH_4Cl	1 in 5 of water. See p. 7.
Ammonium hydrate (or ammonia)	NH_4HO	1 sol. amm. sp. gr. 880, 2 water. (sp. gr. 960). See p. 7.
Ammonium molybdate	$(NH_4)_2MoO_4$	Dissolved in nitric acid. See p. 7.
Ammonium oxalate	$(NH_4)_2C_2O_4$	1 in 25 of water.
Ammonium sulphide	$(NH_4)_2S$	See p. 7.
Ammonium acid sulphite	NH_4HSO_3	See p. 8.
Barium carbonate	$BaCO_3$	See p. 8.
Barium chloride	$BaCl_2$	1 in 10 of water.
Barium hydrate	BaH_2O_2	See p. 8.

Calcium hydrate (or lime)	CaH_2O_2	Saturated solution. See p. 8.
Calcium sulphate . .	$CaSO_4$	Saturated solution. See p. 9.
Copper sulphate . .	$CuSO_4$	1 in 10 of water.
Ferric chloride . .	$FeCl_3$	See p. 9.
Ferrous sulphate . .	$FeSO_4$	Dissolved when required.
Hydrochloric acid, strong	HCl	Pure, sp. gr. 1·16 (containing 32 per cent. HCl). See p. 9
Hydrochloric acid, dilute	HCl	1 strong acid, 2 water.
Hydrocyanic acid . .	HCN	See p. 9.
Hydrofluosilicic acid .	H_2SiF_6	See p. 10.
Hydrosulphuric acid (or sulphuretted hydrogen)	H_2S	Gas and solution. See p. 10.
Indigo solution		Indigo dissolved in strong sulphuric acid and diluted.
Lead acetate . . .	$Pb(C_2H_3O_2)_2$	1 in 10 of water.
Magnesium sulphate .	$MgSO_4$	1 in 10 of water.
Mercuric chloride .	$HgCl_2$	1 in 20 of water.
Nessler's solution . .	K_2HgI_4 in KHO	See p. 11.
Nitric acid, strong .	HNO_3	Pure, sp. gr. 1·45 (containing 77 per cent. HNO_3). See p. 11.
Nitric acid, dilute .	HNO_3	1 strong acid, 4 water.
Nitrohydrochloric acid (or aqua regia) . .	. . . . . .	1 strong nitric acid, 4 strong hydrochloric acid. Mixed as required. See p. 11.
Platinum chloride .	$PtCl_4$	See p. 12.
Potassium bichromate	$K_2CrO_4 . CrO_3$	1 in 10 of water.
Potassium ferricyanide	K_3Cy_6Fe	Dissolved as required.
Potassium ferrocyanide	K_4Cy_6Fe	1 in 10 of water.
Potassium hydrate (or potash)	KHO	See p. 12.

Potassium nitrite . .	KNO_2	See p. 12.
Potassium sulphocyanide	$KCNS$	1 in 10 of water.
Silver nitrate . . .	$AgNO_3$	1 in 20 of water.
Soda chloride . . .	Na_2OCl_2	See p. 13.
Sodium acetate . .	$NaC_2H_3O_2$	1 in 10 of water.
Sodium carbonate .	Na_2CO_3	See p. 13.
Sodium phosphate .	Na_2HPO_4	1 in 10 of water.
Sodium sulphide . .	Na_2S	See p. 13.
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Sulphuric acid, strong	H_2SO_4	Commercial, sp. gr. 1·84 (containing 96 per cent. H_2SO_4).
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Test Papers.

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PREPARATION AND TESTING OF REAGENTS.

DRY REAGENTS.

Microcosmic Salt.

The crystals contain so much water, that it is rather difficult to make a bead with them unless they are previously dried. It is therefore well to dry some of the salt, and keep it for use.

On ignition (in the loop of platinum wire), it is converted into metaphosphate of sodium ($NaPO_3$), water and ammonia being given off.

Potassium and Sodium Carbonate.

This consists of carbonate of potassium and carbonate of sodium, mixed in the proportion of their combining weights.

It is preferred to either of the carbonates alone, because it fuses at a lower temperature. The mixture must be pure. It is best made with the acid carbonates, as these are easily obtained pure, and the excess of carbonic acid does not matter.

Take of

Acid carbonate of potassium, in crystals .	20 grm.
Acid carbonate of sodium, in crystals . .	17 grm.

Powder separately, and mix.

Potassium Cyanide.

Cyanide of potassium is an exceedingly powerful reducing agent in the dry way; indeed it excels in its action almost all other reagents of the same class, and reduces the metals not only from their oxides, but also from their sulphides. The reduction is attended in the former case with formation of cyanate of potassium ($KCNO$), and in the latter case with formation of sulphocyanide of potassium ($KCNS$).

Potassium Acid Sulphate.

Take of

Strong sulphuric acid	49 grm.
Sulphate of potassium, in crystals . . .	87 grm.

Heat the acid in a porcelain dish till it nearly boils, then add the salt gradually. As soon as the fluid is in a state of calm fusion, pour it onto a clean stone.

WET REAGENTS.

Alcohol.

Methylated spirit is sufficiently pure. It contains 10 per cent. of wood spirit. If it leaves a solid residue on evaporation, it should be distilled.

Ammonium Carbonate.

Take of

Carbonate of ammonium, in lumps . .	170 grm.
Solution of ammonia, sp. gr. 960 . .	300 c.c.
Water	to 1 litre.

Put the ingredients into a bottle together, and shake occasionally till the lumps are dissolved.

The reaction which occurs is as follows:—

$$2(NH_4)_2CO_3 \cdot CO_2 + 2NH_4HO = 3(NH_4)_2CO_3 + H_2O.$$

Ammonium Chloride.

If the solution contains iron, add ammonia, allow to stand, filter, and neutralize exactly with hydrochloric acid.

The solution should leave no fixed residue on evaporation, and give no precipitate or color with sulphide of ammonium.

Ammonium Hydrate.

The solution should be colorless, and leave no residue on evaporation. When warmed with an equal volume of lime-water, nothing more than a slight turbidity should be produced (carbonic acid). When supersaturated with nitric acid, no precipitate should be produced either by chloride of barium or nitrate of silver. Sulphuretted hydrogen should not give the slightest color to it.

Ammonium Molybdate.

Take of

Molybdate of ammonium . . .	10 grm.
Solution of ammonia, sp. gr. 960 .	40 c.c.
Strong nitric acid	80 c.c.
Water	80 c.c.

Dissolve the salt in the ammonia by the aid of heat; then pour the solution into the nitric acid and water, which have been previously mixed together.

Ammonium Sulphide.

Take some solution of ammonia (sp. gr. 960), and divide it in half. Saturate one half with sulphuretted hydrogen, and then add the other half.

The reactions which take place are as follows:—

$$NH_4HO + H_2S = NH_4HS + H_2O$$

$$\text{and} \quad NH_4HS + NH_4HO = (NH_4)_2S + H_2O.$$

The solution, which is at first colorless, soon becomes yellow by keeping, from oxidation. The yellow solution deposits sulphur on addition of acids. Yellow sulphide of ammonium may be improvised from the freshly-prepared colorless sulphide by addition of sulphur.

Ammonium Acid Sulphite.

Take charcoal in small pieces, and put it in a flask, add eight times its weight of strong sulphuric acid, and heat the mixture. Pass the evolved gas first through a small empty bottle and then into solution of ammonia (sp. gr. 960) till the ammonia ceases to take it up.

Barium Carbonate.

Dissolve chloride of barium in water, boil the solution, and add excess of carbonate of ammonium. Wash the precipitate by decantation several times, and then on a filter, till the washings are free from chlorine. Finally stir the precipitate up with water to the consistence of cream.

On dissolving in hydrochloric acid, adding excess of sulphuric acid, evaporating the filtrate and igniting, no residue should be left.

Barium Hydrate.

Take of

Witherite, in fine powder .	100 grm.
Lampblack	10 grm.
Rosin	5 grm.

Mix intimately together, and put in a crucible, then heat in a furnace. Powder, boil in an iron pot with water, filter, and allow to stand in a closed vessel. Crystals of $BaH_2O_2 + 8$ aq. will form. Drain them in a covered funnel, and dry between blotting paper. Dissolve 1 part of the crystals in 20 parts of water, and filter.

The solution is generally called baryta water. On mixing with excess of sulphuric acid, and filtering, the filtrate should leave no fixed residue on evaporation.

Calcium Hydrate.

Take of

Lime	10 grm.
Water	1 litre.

Put into a bottle together. When the lime has slaked shake the bottle for two or three minutes. After twelve hours the excess of lime will have subsided, and the solution may be drawn off with a syphon.

The solution is commonly called lime water. It contains about 1 part of lime in 700 parts.

Calcium Sulphate.

Take of

Gypsum, in powder . . .	10 grm.
Water	1 litre.

Put into a bottle and shake occasionally for some time. Allow the undissolved portion to subside, and decant the clear fluid.

The solution contains about 1 part of the salt in 400 parts.

Ferric Chloride.

Dilute some strong hydrochloric acid with ten parts of water, and warm the mixture in a flask with an excess of iron wire. As soon as the hydrogen ceases to escape, filter, and conduct chlorine into the solution with frequent agitation. Continue passing the chlorine till a drop of the fluid taken out gives no precipitate with ferricyanide of potassium. Finally heat till the excess of chlorine is driven off.

Hydrochloric Acid.

Hydrochloric acid must be colorless, and leave no residue on evaporation. If it turns yellow on evaporation, ferric chloride is present. It must not impart a blue color to a solution of iodide of potassium mixed with starch solution (chlorine or ferric chloride), nor decolorize indigo-solution (chlorine), nor decolorize a fluid made faintly blue with iodide of starch (sulphurous acid). When diluted, it should give no precipitate with chloride of barium (sulphuric acid). Hydrosulphuric acid should produce no alteration (arsenic). On neutralization with ammonia, and addition of sulphide of ammonium, no change should take place (iron, thallium).

Hydrocyanic Acid.

Take of

Ferrocyanide of potassium	150 grm.
Strong sulphuric acid	50 c.c.
Water	500 c.c.

Mix the acid with the water, and allow to cool, then pour the mixture on the salt in lumps contained in a litre flask. Distil with a Liebig's condenser. Put 50 c.c. of water in the receiver. When 300 c.c. have been obtained, stop the operation. Put two or three drops of dilute sulphuric acid into the product.

Hydrofluosilicic Acid.

Take of

Sand	100 grm.
Fluor spar, in powder . . .	100 grm.
Strong sulphuric acid. . . .	350 c.c.
Water	400 c.c.

Wash the sand from dust, and dry thoroughly. Mix it with the fluor spar, also dry, and then with the sulphuric acid in a stone-ware bottle of one and a half or two litres capacity. Place the bottle in an iron pot filled with sand. Connect the neck of the bottle with a small empty bottle, and the latter again with a wide tube bent twice at right angles. Join the outer end of the tube to a funnel by means of Indian rubber, and immerse the mouth of the funnel just into the water. Towards the end of the process the heat requires to be pretty strong. Fluoride of silicon comes over, which decomposes with the water, forming hydrated silica and hydrofluosilicic acid. The hydrate of silica would at once stop up the mouth of the delivery tube if it dipped directly in the water. When the evolution of gas has ceased, throw the gelatinous paste upon a linen cloth, squeeze the fluid through, and afterwards filter it.

Hydrofluosilicic acid must produce no precipitate in solutions of salts of strontium (sulphuric acid).

Hydrosulphuric Acid.

THE GAS.

Hydrosulphuric acid is made by the action of dilute sulphuric acid upon sulphide of iron. In large laboratories it may be stored in a gasometer, and supplied like coal gas. In small laboratories it is made when required, from sulphide of iron and dilute sulphuric acid.

When the apparatus is done with, the evolution-bottle should be washed out, or the ferrous sulphate produced may crystallize in a mass and be difficult to remove. The sulphide of iron which remains unacted on should be returned to the bottle, as it will do for next time.

THE SOLUTION.

The solution is prepared by conducting the gas into water until the latter is saturated. The water may be considered to be saturated when, on closing the mouth of the bottle with the thumb, and shaking it a little, a pressure is felt from within.

In contact with air, the solution decomposes gradually, the hydrosulphuric acid being converted into water, sulphur, and a little sulphuric acid. Therefore, if the solution is required to be kept for any length of time, the bottle should be full and well closed. The solution may be kept for months if the bottle is inverted in water.

Nessler's Solution.

Take of

Iodide of potassium	3·5 grm.
Mercuric chloride	1·6 grm.
Water	40 c.c.
Solution of potash	A sufficiency.

Dissolve the iodide of potassium in 10 c.c. of water. Dissolve the mercuric chloride in 30 c.c. of water. Add the latter solution to the former gradually, till a permanent precipitate is produced. Then add solution of potash till the fluid measures 100 c.c., and filter.

Nitric Acid.

Nitric acid should be colorless and leave no residue on evaporation. When diluted with plenty of water, it should give no precipitate with nitrate of silver, or with chloride of barium.

Nitrohydrochloric Acid.

Nitric acid and hydrochloric acid decompose each other, the decomposition mostly resulting, as Gay-Lussac showed, in the formation of two compounds, $NOCl_2$ and $NOCl$, and of free chlorine and water. Thus

$$HNO_3+3HCl=NOCl_2+Cl+2H_2O,$$

and $$HNO_3+3HCl=NOCl+Cl_2+2H_2O.$$

The presence of free chlorine makes aqua regia the most powerful solvent for metals, with the exception of those which form insoluble compounds with chlorine. Aqua regia serves principally for the solution of gold and platinum, and for the decomposition of various sulphides, such as cinnabar, pyrites, &c.

Platinum Chloride.

Weigh out 25 grm. of platinum clippings, and boil them with strong nitric acid for some time; then pour away the acid and wash the clippings with a little water. Now digest at a gentle heat with 500 c.c. of aqua regia, adding more aqua regia if necessary, until the metal is dissolved. Transfer the solution to a porcelain dish, and evaporate on the water bath to the consistence of syrup; add some strong hydrochloric acid, mix, and evaporate again. The addition of hydrochloric acid and second evaporation are to remove nitric acid. Lastly, dissolve in water to 500 c.c.

Platinum residues may be worked up as follows:—Evaporate altogether to dryness, ignite in an open clay crucible, to decompose the double salts and burn off organic matter, boil with hydrochloric acid, then with water, then with nitric acid, and wash with water. The pure platinum thus obtained is treated as above.

Chloride of platinum, when evaporated to dryness on a water bath, should leave a residue completely soluble in alcohol.

Potassium Hydrate.

Take of

Potash	200 grm.
Water	to 1 litre.

Dissolve. Allow to settle, and draw off the clear fluid.

The potash of commerce is generally impure. Where a pure caustic alkali is required, the soda prepared from sodium seems to recommend itself.

Potassium Nitrite.

In an iron pan fuse 1 part of nitre, add 2 parts of lead, and keep stirred with an iron rod. Even at a low red heat the lead becomes for the most part oxidized and converted into a yellow powder. To oxidize the remainder, the heat is increased to visible redness, and maintained at that point for half an hour. Allow to cool, treat with cold water, filter and pass carbonic acid through the filtrate. This precipitates almost the whole of the lead in solution, the remainder is removed with sulphuretted hydrogen. Evaporate the clear fluid to dryness, finally with stirring, and fuse in order to destroy any hyposulphite of potassium. Dissolve 1 part in 2 parts of water, and neutralize cautiously with acetic acid.

Soda Chloride.

Take of

Chloride of lime	100 grm.
Water	1 litre.
Solution of commercial carbonate of sodium	A sufficiency.

Put the chloride of lime into a bottle with the water, and digest for three hours, shaking several times. Allow to stand, draw off the clear solution from the undissolved residue, and add carbonate of sodium till it ceases to produce a precipitate—this may be known by filtering a small quantity, and testing the filtrate with carbonate of sodium. Finally, allow to settle, and draw off the clear fluid for use.

Sodium Carbonate.

Dissolve 1 part of the anhydrous salt, or $2\frac{1}{2}$ parts of the crystals, in 7 parts of water.

The solution, after acidification with nitric acid, should give no precipitate with chloride of barium or nitrate of silver.

Sodium Sulphide.

Take some solution of soda (hydrate of sodium) and divide it in half. Saturate one half with sulphuretted hydrogen and add the other half.

Stannous Chloride.

Take 50 grm. granulated tin,[*] and dissolve it by digesting with strong hydrochloric acid in a flask. Mix the solution

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[*] Made by melting grain tin in a porcelain dish, and pouring it into cold water.

with dilute hydrochloric acid till it measures 1 litre, and keep it in a bottle with some pieces of tin.

This solution is liable to oxidation.

Sulphuric Acid Dilute.

Take of

Strong sulphuric acid. . . .	200 c.c.
Water	1 litre.

Add the acid to the water with stirring. Allow the sulphate of lead to settle, and decant.

Sulphuric Acid Pure.

Take of

Strong sulphuric acid	1000 grm.
Sulphate of ammonium	3 grm.
Binoxide of manganese, in powder .	5 grm.

Put the acid in a porcelain dish, add the sulphate of ammonium, and heat till copious fumes of sulphuric acid escape. This is done to destroy any oxides of nitrogen. After cooling, add the manganese, and heat to boiling with stirring, in order to convert any arsenious acid into arsenic acid. When cool, pour off the clear fluid into a retort of a litre capacity, and distil. The neck of the retort must reach so far into the receiver that the distillate may drop directly into its body. The receiver should not be cooled with water. To prevent the neck of the retort from touching the receiver, some long asbestos may be used. When about 10 c.c. have been drawn over, change the receiver, and distil off three-fourths of the contents of the retort. This method depends upon the fact that sulphuric acid containing arsenic in the form of arsenic acid, yields an arsenic-free distillate.

Pure sulphuric acid should leave no residue upon evaporation. When solution of ferrous sulphate is poured upon it in a test-tube, no color should mark the plane of contact of the two fluids (nitric acid, peroxide of nitrogen). When diluted with twenty parts of water, it must not impart a blue tint to a solution of iodide of potassium mixed with a solution of starch (peroxide of nitrogen). Mixed with arsenic-free zinc and water, it must yield hydrogen gas, which, on being passed through a red-hot tube, must not deposit a trace of arsenic.

When hydrochloric acid is added to it in a test tube, no turbidity should mark the plane of contact (lead).

Water, Distilled.

A copper still and tin worm should be used. About three-fourths of the water should be drawn over.

Zinc, Granulated.

Melt scraps of zinc, and pour in a thin stream into a pail of cold water.

The zinc used in testing for arsenic must of course be free from that metal. Zinc which contains arsenic must be absolutely rejected for this purpose, as it cannot be purified by any known practical process.

TEST PAPERS.

The paper should be prepared as below, and cut into slips of 1 by 5 cm.

Litmus Paper.

Digest 20 grm. litmus, with 100 c.c. water for some days, shaking occasionally; then filter. To the filtrate add slight excess of nitric acid and boil; then neutralize exactly with potash. Now make a weak solution of gelatine by boiling 1 part of isinglass with 50 parts of water, draw white blotting paper through this and hang it up to dry. When dry, paint one side with the above solution of litmus.

This paper has a neutral tint, and is very delicate.

Turmeric Paper.

Digest one part of bruised turmeric at a gentle heat, with 6 parts of a mixture of equal parts of spirit and water. Filter, and paint white sized paper on one side with this tincture.

APPARATUS AND MANIPULATION.

LIST OF APPARATUS.

Basket for test-tubes.
Beakers.
Berlin crucibles.
Berlin dishes.
Blowpipe.
Boiling tubes (large test-tubes, 8 by 1 inch).
Bunsen lamp.
Charcoal.
Corks.
Crucible tongs.
Files, rat-tail and triangular.
Filters, cut, 2, 3¾, 4½, 5½ inches diameter.
Filter-stand.
Funnels, 1, 2, 2½, 3 inches diameter.
Glass rods.
Glass tubing, soft and hard.
Pestle and mortar, porcelain.
Platinum crucible and cover.
Platinum wires fixed in glass tubes.
Sulphuretted hydrogen apparatus.
Test-tubes.
Test-tube holder, wooden.
Test-tube stand.
Tobacco-pipe triangles.
Triangular tripod.
Vulcanite spatula (7 inches long).
Vulcanized tubing.
Wash-bottle.
Watch-glasses.
Water bath, copper. Hemispherical, 7 inches diameter across the top, with set of rings.
Wire gauze, iron,* 6 inches square, 6 holes in inch linear.

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* This is laid on the top of the tripod, and serves to support beakers,

Agate mortar.
Platinum spatula.
Steel mortar.
The last three are unnecessary for beginners.

MANIPULATION.

The Blowpipe.

In testing substances before the blowpipe we use ignition tubes, charcoal, and platinum wire.

The ignition tubes* are made by cutting some hard glass tubing of 5 mm. bore into lengths of 12 cm., drawing them out in the middle and sealing them. Each length thus makes two tubes sealed at one end.

The charcoal should be free from fissures. It should be sawn into pieces about 10 cm. long, and these again should be sawn in half longitudinally. Before use a small semicylindrical cavity should be scooped out with a knife on the flat side at one end. Minute metallic globules are best detected by cutting out the part where they are, pounding it in a mortar, and washing repeatedly by decantation. The metal being heavy will finally remain by itself.

Platinum wire should not be too thin or it will soon break, it should be cut into lengths of 5 cm. and fused into pieces of glass tube a little longer. Borax and microcosmic beads are made by bending the end of the wire into a loop, igniting it, dipping it in the salt, and igniting again till a clear bead is obtained. This is then dipped in the substance to be tested. Old beads are loosened by igniting and dipping in cold water, after which they can readily be removed by the fingers. In testing the coloring action of a substance on flame, a *clean* platinum wire is dipped into it, and then held in the blowpipe (or Bunsen) flame; sometimes the substance is moistened with strong hydrochloric acid to increase its coloring effect. Platinum wire may be cleaned by being dipped red hot into strong hydrochloric acid repeatedly.

&c., during heating. It does not *protect* the vessels much, and if the flame is high at first, glass is apt to crack. A safer support consists of the so-called gauze-plate, which is made by backing a piece of wire-gauze with tin plate. The piece of tin should be a little larger than the piece of gauze, so that its edges may be turned over to connect the two.

* Ignition signifies heating to redness. An ignition tube is a tube for heating substances to redness in.

For operations which last some time, Herapath's blowpipe is convenient. For fusions in crucibles and glass working, we may use a large Herapath with bellows worked by the foot.

Powdering.

A porcelain mortar is generally used for this purpose. Very hard minerals must be broken up with a hammer, then reduced to a coarse powder in a steel crushing mortar, and finally rubbed down in an agate mortar. In powdering minerals, a little sieve made of a piece of muslin and a chip box with both the ends pushed out will be found of service.

Precipitation.

Precipitation signifies the sudden reduction of a dissolved body to the solid state, no matter whether it sinks to the bottom or rises to the top, or remains suspended in the fluid. The solid body separated is called the precipitate, and the substance which acts as the immediate cause of separation is called the precipitant. The terms turbid and cloudy are employed to designate the state of a fluid which contains a precipitate so finely divided and so inconsiderable in amount, that the suspended particles although impairing the transparency of the fluid yet cannot be clearly distinguished.

Separation of Precipitates from Fluids.

Under this head we have the following operations :—Filtration, Decantation, Washing.

Filtration.

Filtration is the passing of a liquid through a porous substance, or filter.

The filter consists of a properly folded piece of filter paper which is placed in a funnel. The paper should be cut into a circle, then folded into a quadrant and opened into a cone. The funnel either rests in a test-tube or wide-mouthed bottle,*

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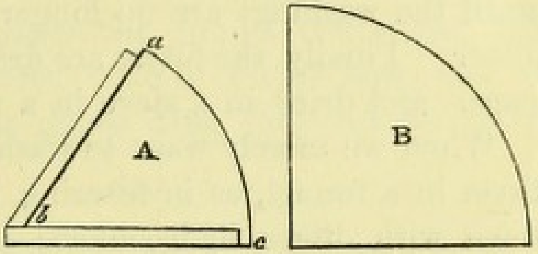
* A few eight-ounce white round bottles will be found useful for filtering larger quantities of fluid, or for supporting a filter during the washing of a precipitate. They are cheaper and less fragile than beakers. Of course they are of no use for boiling.

or is supported in a filter-stand. The funnel should be of such a shape that the paper may fit closely to its sides. The filtered fluid is called the filtrate. If the filtrate is at first turbid it is returned to the filter.

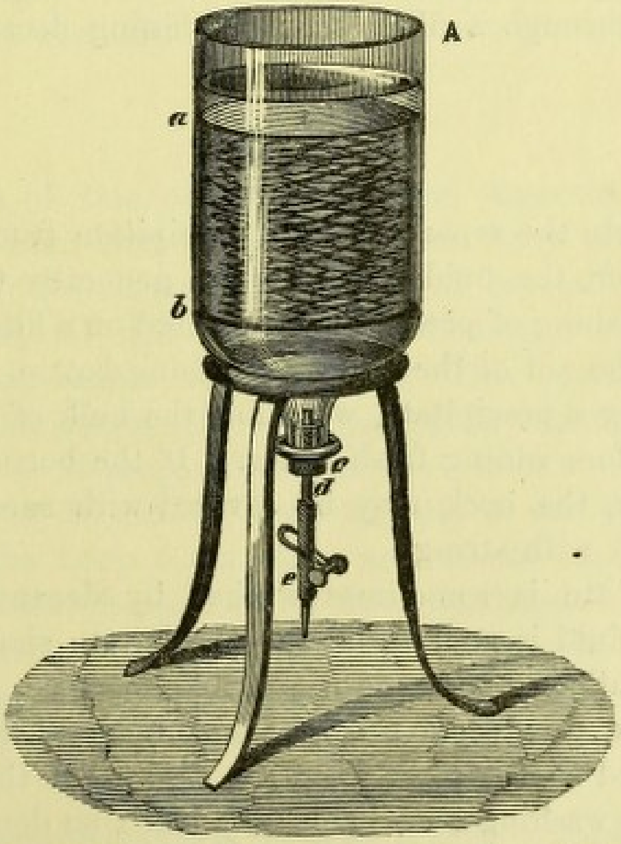
Note on filters.—Round filter papers can be bought already cut, or may be made at home from fine white blotting paper with the aid of Mohr's filter patterns (fig. 1.) This little apparatus is made of tin plate, and consists of two parts. B is a quadrant fitting in A, whose straight edges are turned up, and which is slightly larger than B. The sheets of filter paper are first cut up into squares, which are folded in quarters and placed in A; then B is placed on the top, and the free edge of the paper is cut off with scissors. Three pairs of these patterns may be procured of 48, 58, and 68 mm. radius respectively.

Fig. 1.



If filtering paper is to be used for very delicate analysis it requires washing with acid and water. For this purpose the apparatus shown in the figure will be found convenient. A is

Fig. 2.



a bottle with the bottom out; *a* and *b* are glass plates; between these lie the filters which have been previously cut and folded. The bottle is filled with dilute hydrochloric acid, in which the filters are allowed to soak for twelve hours; the acid is then run off and replaced by water. After an hour this is replaced by fresh water, and so on till the washings are barely acid. The washing is finished with distilled water; it is continued until the washings are no longer rendered turbid by nitrate of silver. Finally, the filters are drained, turned out onto blotting paper, and dried in a sieve in a warm place.

When we merely want to wash two or three filters, we place them in a funnel, as in filtering, one inside the other, moisten them with dilute hydrochloric or nitric acid, and then wash them.

Decantation.

Decantation is the pouring of a fluid from one vessel to another. It is used instead of filtration sometimes, when the precipitate readily falls to the bottom.

Occasionally we pour off a supernatant fluid, which is not quite clear, through a filter, thus combining decantation with filtration.

Washing.

To complete the separation of a precipitate from extraneous soluble matter, the fluid adhering has generally to be washed out. The washing of precipitates collected on a filter, is usually effected by the aid of the familiar washing-bottle.

In washing a precipitate, we allow the bulk of the fluid to drain out before adding fresh water. If the bottle is used for boiling water, the neck may be covered with several folds of flannel bound with string.

A precipitate is sometimes washed by decantation. The supernatant fluid is poured off, the precipitate shaken up with fresh water, allowed to settle again, and so on.

If we have to wash a precipitate completely, the washings must be tested to see when they are free from the substance which we are washing out. This may often be done by evaporating a drop on a platinum knife, or by mixing a few drops with a drop of nitrate of silver on a watch-glass, &c.

Drying of Precipitates.

The greater portion of water adhering to a precipitate that has been collected on a filter may be removed by taking the filter out of the funnel and pressing it between blotting-paper, at first lightly, then heavily, changing the blotting-paper. A hot brick will also be found useful to spread out the filter upon. Another way is to place the funnel containing the precipitate in a hollow truncated cone of tin or zinc, and to support the latter on a wire gauze over the Bunsen lamp turned very low.*

Incineration of Filters.

When a small precipitate has to be examined for a fixed metal, it is frequently convenient to incinerate the filter containing it. This is done by drying the filter, rolling it up, inclosing it in two or three coils of platinum wire, and holding it in the Bunsen flame. The lamp should be held sideways, and a watch-glass or dish should be placed under the burning filter to catch the ash.

Dialysis.

By means of this operation, we can separate the class of crystalloids from that of colloids when they are in solution together. To the former class belong all crystallizable bodies; to the latter, all non-crystallizable bodies, such as gelatin, gum, caramel, tannin, albumen, &c. This operation is often of considerable service in chemico-legal investigations.

The dialyser is prepared as follows:—take a disk of parchment paper 30–35 cm. in diameter, wet it, and spread it over a gutta-percha hoop 5 cm. deep, and 20–25 cm. in diameter; then slip another hoop over, or bind the paper in some other way (it should not be too firmly secured). The paper must not be porous. Its soundness may be tested by sponging the upper side with water, and observing whether wet spots show

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* Under these circumstances the flame is liable to descend. This may be obviated by laying a piece of coarse wire gauze an inch square on the top of the lamp.

on the other side. Defects may be remedied by applying liquid albumen, and then coagulating it by heat.

The dialyser being thus prepared, the liquid to be dialysed is poured into it so as to cover the paper to a depth of not more than 12 mm. The apparatus is then floated in a pan, containing about four times as much water as the fluid to be dialysed amounts to. After twenty-four hours, half or three-fourths of the crystalloids will be found in the external water, while the colloids remain in the dialyser—at most only traces pass into the external fluid. By floating the dialyser successively on fresh supplies of water, the whole of the crystalloids may finally be removed.

Evaporation.

Evaporation signifies the conversion of a liquid into vapor. The process is usually conducted in a porcelain dish, which is heated either directly over the flame or on a water-bath, according to circumstances.

Note.—Porcelain and glass are slightly attacked by the protracted action of fluids at high temperatures. Evaporating liquids are therefore more or less contaminated by the constituents of the vessels, which in delicate analyses becomes an annoyance. For details on this subject I refer to the " Quantitative Analysis." However, it may be taken as a rule that alkaline fluids should never be evaporated in glass.

Platinum Crucibles.

The following substances must not be put into platinum crucibles—viz., nitrates, hydrates, and cyanides of potassium and sodium, metals, sulphides, readily reducible metallic oxides, organic salts of heavy metals, phosphates in presence of organic compounds.

Platinum crucibles may be cleaned by rubbing them with a little moistened pumice powder.* The platinum must not be rubbed too much with this material, as it removes the metal. If the stain still remains, fuse some acid sulphate of potassium in the crucible for some minutes at a *moderate* temperature.

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* This is also useful for cleaning porcelain and glass from dirt which is not readily removable by acids.

REACTIONS AND SEPARATION.

REACTIONS OF THE METALS.

DIVISION OF THE METALS INTO GROUPS.

If to a solution containing all the common metals we add hydrochloric acid, silver, monad mercury, and lead are precipitated.

On filtering and passing sulphuretted hydrogen through the fluid, dyad mercury, bismuth, copper, cadmium, tin, antimony, and arsenic are precipitated.

On filtering again and adding chloride of ammonium to prevent the precipitation of the magnesium, ammonia to neutralize the excess of hydrochloric acid, and lastly, sulphide of ammonium, nickel, cobalt, iron, manganese, zinc, chromium, and aluminium are precipitated.

On filtering again and adding carbonate of ammonium, barium, strontium, and calcium are precipitated.

The filtrate from the last precipitate contains the rest of the metals — viz., magnesium, potassium, sodium, and ammonium.

On these facts the division of the metals into groups is based as follows :—

Group I.

METALS PRECIPITATED BY HYDROCHLORIC ACID (AS CHLORIDES).

Silver.
Monad mercury.
Lead.

GROUP II.

METALS NOT PRECIPITATED IN PREVIOUS GROUP, BUT THROWN DOWN BY HYDROSULPHURIC ACID IN PRESENCE OF A MODERATE EXCESS OF HYDROCHLORIC ACID (AS SULPHIDES).

Division I.

The sulphides insoluble in sulphide of ammonium.

Dyad mercury.
Bismuth.
Copper.
Cadmium.

Division II.

The sulphides soluble in sulphide of ammonium.

Tin.
Antimony.
Arsenic.

GROUP III.

METALS NOT PRECIPITATED IN PREVIOUS GROUPS, BUT THROWN DOWN BY AMMONIA AND SULPHIDE OF AMMONIUM IN PRESENCE OF CHLORIDE OF AMMONIUM.

(As sulphides.)
Nickel.
Cobalt.
Iron.
Manganese.
Zinc.

(As hydrates.)
Chromium.
Aluminium.

GROUP IV.

METALS NOT PRECIPITATED IN PREVIOUS GROUPS, BUT THROWN DOWN BY CARBONATE OF AMMONIUM IN PRESENCE OF CHLORIDE OF AMMONIUM (AS CARBONATES).

Barium.
Strontium.
Calcium.

GROUP V.

METALS NOT PRECIPITATED IN PREVIOUS GROUPS.

Magnesium.
Potassium.
Sodium.
Ammonium.

THE REACTIONS OF THE METALS.

GROUP I.

SILVER, MONAD MERCURY, LEAD.

Silver.

DRY REACTION.

1. Mixed with carbonate of sodium and heated on *charcoal* before the blowpipe; compounds of silver give white brilliant malleable metallic globules.

WET REACTIONS.

2. *Hydrochloric acid** produces a white curdy precipitate of chloride of silver ($AgCl$). In very dilute solutions this reagent simply causes a bluish-white opalescence at first; but after long-standing the chloride collects at the bottom of the vessel. By the action of light the chloride first acquires a violet color, and ultimately turns black—these changes are accompanied by loss of chlorine. Chloride of silver is insoluble in nitric acid, but dissolves readily in ammonia—on addition of an acid to this solution the chloride of silver reprecipitates. Concentrated hydrochloric acid and concentrated solutions of potassium and sodium chlorides, dissolve chloride of silver to a very perceptible extent, more particularly when heated; but the dissolved chloride separates again on dilution. Upon exposure to heat chloride of silver fuses without decomposition, giving upon cooling a transparent horny mass.

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* As a test we use the dilute acid.

3. *Hydrosulphuric acid*[*] precipitates black sulphide of silver (Ag_2S), which is insoluble in dilute acids, alkalies, alkaline sulphides, and cyanide of potassium. Boiling nitric acid dissolves this precipitate readily, with separation of sulphur.

4. *Sulphide of ammonium* produces the same precipitate.

5. *Potash* precipitates oxide of silver (Ag_2O) as a greyish brown powder, insoluble in excess of the precipitant, soluble in ammonia.

6. *Ammonia*, added in small quantity to neutral solutions, throws down the oxide, which readily dissolves in excess of the precipitant.

Monad Mercury.

DRY REACTIONS.

1. The compounds volatilize when heated in an *ignition tube*.

2. If a mixture of an anhydrous compound of mercury and dry *carbonate of sodium*[†] is introduced into an *ignition tube*, covered with a little dry carbonate of sodium, and then heated in the blowpipe flame, the mercury sublimes, forming a grey ring. The bottom of the tube may be broken off by the aid of a file, and then the sublimate may be united into distinct globules by rubbing.

WET REACTIONS.

3. *Hydrochloric acid* precipitates mercurous chloride ($HgCl$) as a fine white powder. Cold hydrochloric acid and cold nitric acid fail to dissolve the precipitate; it dissolves, however, upon long-continued boiling with these acids, but only with difficulty, being converted by the former into mercuric chloride and metallic mercury, which separates, and by the latter into mercuric chloride and mercuric nitrate. Aqua regia and chlorine-water dissolve the mercurous chloride readily, converting it into mercuric chloride. Ammonia and potash decompose mercurous chloride, the former transforms it into NH_2Hg_2Cl, and the latter into mercurous oxide.

4. *Hydrosulphuric acid* produces a black precipitate of

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[*] As a test we generally use the solution.

[†] The carbonate of sodium must be thoroughly dried by ignition on a broken piece of porcelain.

mercurous sulphide (Hg_2S), which is insoluble in dilute acids, sulphide of ammonium, and cyanide of potassium. Sulphide of sodium (Na_2S) in presence of caustic soda, dissolves this precipitate with separation of metallic mercury; sulphide of sodium containing excess of sulphur dissolves it without separation of metallic mercury; the solutions contain mercuric sulphide. When boiled with concentrated nitric acid, mercurous sulphide gives up part of its mercury, and a white compound [$Hg(NO_3)_2 + 2HgS$] is formed. Mercurous sulphide is readily dissolved by aqua regia.

5. *Sulphide of ammonium* produces the same precipitate.

6. *Potash* produces a black precipitate of mercurous oxide (Hg_2O), insoluble in excess of the precipitant.

7. *Ammonia* produces a black precipitate of a complicated composition, insoluble in excess of the precipitant.

8. If a drop of a mercurous solution is put on a clean plate of *copper*, and washed off after some time, a spot will be produced which, on being gently rubbed, will appear bright and shining like silver. The application of a gentle heat to the copper causes the mercury to volatilize, and thus removes the silvering.

9. *Stannous chloride* produces a grey precipitate of metallic mercury. By decanting the supernatant fluid and boiling the deposit with hydrochloric acid, distinct globules will be obtained.

Lead.

DRY REACTION.

1. Mixed with carbonate of sodium, and heated on *charcoal* before the blowpipe, compounds of lead give malleable metallic globules.

WET REACTIONS.

2. *Hydrochloric acid* produces in somewhat strong solutions a heavy white precipitate of chloride of lead ($PbCl_2$), soluble in a large amount of water, especially when heated. The chloride of lead is converted by ammonia into a basic salt ($PbCl_2 . 3PbO + H_2O$), also a white powder, but almost absolutely insoluble in water. In dilute nitric and hydrochloric acids, chloride of lead is more difficultly soluble than in water.

3. *Hydrosulphuric acid* produces a black precipitate of

sulphide of lead (PbS), which is insoluble in cold dilute acids, alkalies, alkaline sulphides, and cyanide of potassium. It is decomposed by hot nitric acid. If the acid is dilute the whole of the lead is obtained in solution as nitrate, and sulphur separates; if the acid is fuming, the sulphide of lead is converted completely into sulphate (insoluble). In solutions containing a large quantity of free hydrochloric acid, hydrosulphuric acid sometimes produces a red precipitate; this consists of chloride and sulphide of lead; it is converted into the black sulphide by excess of hydrosulphuric acid.

4. *Sulphide of ammonium* produces the same precipitate.

5. *Potash* throws down a white precipitate of basic salt, which is soluble in excess of the precipitant.

6. *Ammonia* throws down a white precipitate of basic salt, which is insoluble in excess of the precipitant. If the ammonia is free from carbonic acid a precipitate is not at once produced in acetate of lead, owing to the formation of a soluble basic acetate.

7. *Carbonate of sodium* produces a white precipitate of basic carbonate of lead (*e.g.* $6PbCO_3 . PbH_2O_2$) which is not quite insoluble in excess of the precipitant, especially on warming, but is insoluble in cyanide of potassium.

8. *Sulphuric acid* produces a white precipitate of sulphate of lead ($PbSO_4$), nearly insoluble in water and dilute acids. From dilute solutions, especially from such as contain much free acid, the sulphate of lead may not precipitate until after a long time. It is advisable to add a considerable excess of dilute sulphuric acid, sulphate of lead being more insoluble in dilute sulphuric acid than in water. The separation of small quantities of sulphate of lead is best effected by evaporating, after the addition of the sulphuric acid, on a water-bath, and then treating the residue with spirit of wine. Sulphate of lead is slightly soluble in strong nitric acid; it dissolves with some difficulty in boiling strong hydrochloric acid, more readily in potash. It dissolves also pretty readily in the solutions of some of the salts of ammonium, particularly in acetate of ammonium; dilute sulphuric acid reprecipitates it from these solutions.

9. *Bichromate of potassium* produces a yellow precipitate of chromate of lead ($PbCrO_4$), readily soluble in potash, difficultly soluble in dilute nitric acid.

Group II. Division I.

DYAD MERCURY. BISMUTH. COPPER. CADMIUM.

Dyad Mercury.

DRY REACTIONS.

The same as those of monad mercury.

WET REACTIONS.

1. *Hydrosulphuric acid* if added in small quantity produces, after shaking, a perfectly white precipitate, a larger quantity causes the precipitate to turn yellow, and an excess produces a black precipitate of mercuric sulphide (HgS). This peculiarity is characteristic of mercuric solutions. The white precipitate consists of mercuric sulphide, with the undecomposed salt; as more sulphide is formed so the color of the precipitate deepens. Sulphide of ammonium only dissolves traces of mercuric sulphide, the least mercury is dissolved when the sulphide is digested hot with yellow sulphide of ammonium. Potash and cyanide of potassium do not dissolve mercuric sulphide. It is altogether insoluble in hydrochloric acid and in nitric acid, even upon boiling. It dissolves completely in sulphide of sodium in presence of soda; it is readily dissolved by aqua regia.

2. *Sulphide of ammonium* acts in the same way.

3. *Potash* added in excess produces a yellow precipitate of mercuric oxide (HgO), insoluble in excess of the precipitant. In very acid solutions the precipitation is far from complete. In presence of salts of ammonium, a white precipitate, similar to that produced by ammonia alone, is thrown down.

4. *Ammonia* produces a white precipitate. In the case of mercuric chloride this consists of $N_2H_4Hg_2Cl_2$.

5. *Metallic copper* reacts with mercuric salts in the same way as with mercurous salts.

6. If a little *galvanic element*, made out of a slip of platinum foil and a slip of tin foil, joined at one end with a wooden clamp and otherwise apart from each other, is introduced into a mercuric solution, acidified with hydrochloric acid, all the mercury will gradually be precipitated by preference upon the platinum. On removing the foils, drying and heating

strongly in an ignition tube, globules of mercury will be obtained, which may be more distinctly seen under the microscope. On heating this mercury with a fragment of iodine, it will be converted into red mercuric iodide.

7. *Stannous chloride* added in small quantity to a mercuric solution containing hydrochloric acid, produces a white precipitate of mercurous chloride, which turns grey on addition of excess of the reagent, being converted into metallic mercury. When the grey precipitate has subsided, the supernatant fluid may be poured off, and the residue boiled with hydrochloric acid and a little stannous chloride, in order to obtain the mercury in the form of visible globules.

Bismuth.

DRY REACTION.

1. Compounds of bismuth mixed with carbonate of sodium and heated on *charcoal* in the inner flame of the blowpipe, yield brittle metallic globules.

WET REACTIONS.

2. *Hydrosulphuric acid* produces a black precipitate of sulphide of bismuth (Bi_2S_3), which is insoluble in dilute acids, alkalies, alkaline sulphides, and cyanide of potassium, but is readily dissolved by boiling nitric acid.

3. *Sulphide of ammonium* produces the same precipitate.

4. *Potash* throws down hydrate of bismuth, as a white precipitate insoluble in excess of the precipitant.

5. *Ammonia* throws down the same precipitate, also insoluble in excess of the precipitant.

6. *Carbonate of ammonium* throws down a basic carbonate of the composition $Bi_2O_3 . CO_2$, as a white bulky precipitate, insoluble in excess of the precipitant and also in cyanide of potassium.

7. *Bichromate of potassium* precipitates a chromate of bismuth ($Bi_2O_3 . 2CrO_3$) as a yellow powder. This substance differs from chromate of lead in being readily soluble in dilute nitric acid and insoluble in potash.

8. *Dilute sulphuric acid* fails to precipitate solutions of bismuth. On evaporating with an excess of sulphuric acid on

the water-bath to dryness, a white saline mass is left, which readily dissolves in water acidified with sulphuric acid. After standing, perhaps, for several days, a basic sulphate of bismuth ($Bi_2O_3 . SO_3 + 2aq.$) separates from this solution in white microscopic needles, which are soluble in nitric acid.

9. *Water* precipitates basic salts if there is not too much free acid present. This reaction is the most sensitive with the chloride of bismuth, as the basic chloride ($BiCl_3 . Bi_2O_3$) is almost absolutely insoluble in water. Hence when water fails to precipitate a nitric acid solution of bismuth from the presence of too much free acid, a precipitate will generally appear immediately upon addition of chloride of ammonium. Presence of tartaric acid does not interfere with the precipitation of solutions of bismuth by water.

Copper.

DRY REACTIONS.

1. If copper, either in the metallic state or in combination, is held in the *Bunsen flame,* the latter acquires a magnificent green color. Addition of hydrochloric acid considerably heightens the beauty of this very delicate reaction, as it occasions the appearance of an azure colour.

2. A mixture of a compound of copper with carbonate of sodium and cyanide of potassium, exposed on *charcoal* to the inner blowpipe flame, gives metallic copper.

3. The *borax* bead, held in the outer blowpipe flame, is colored by compounds of copper. The bead is green while hot, and blue when cold.

WET REACTIONS.

4. *Hydrosulphuric acid* produces a brownish-black precipitate of sulphide of copper (CuS). It is insoluble in dilute acids and caustic alkalies. Hot sulphide of sodium fails to dissolve it, or dissolves it only to a very trifling extent; it is a little more soluble in sulphide of ammonium, especially when the latter is yellow and hot. Sulphide of copper is readily dissolved by boiling nitric acid, but it is entirely unacted upon by boiling dilute sulphuric acid. It dissolves completely in cyanide of potassium.

5. *Sulphide of ammonium* produces the same precipitate.

6. *Potash** produces a light blue bulky precipitate of hydrate of copper (CuH_2O_2). On boiling in presence of excess of potash, the precipitate turns black immediately, at the same time becoming denser. This change takes place in the cold after some time, if the solution is very concentrated. It depends upon the conversion of the CuH_2O_2 into $3CuO . H_2O$.

7. *Carbonate of sodium** produces a greenish blue precipitate of basic carbonate of copper ($CuCO_3 . CuH_2O_2$), which, upon boiling, changes to brownish-black hydrated oxide. It dissolves in ammonia to an azure, and in cyanide of potassium to a colorless fluid.

8. *Ammonia*, added in small quantity to neutral solutions, produces a greenish-blue precipitate of a basic salt. This precipitate redissolves upon further addition of the reagent to a clear fluid of a magnificent azure color. The blue color is perceptible in very dilute solutions. Potash produces in such blue solutions in the cold, after the lapse of some time, a precipitate of blue hydrate of copper; but upon boiling, this reagent precipitates the whole of the copper as black hydrated oxide.

9. *Carbonate of ammonium* gives the same reactions as ammonia.

10. *Ferrocyanide of potassium* produces a reddish-brown precipitate of ferrocyanide of copper (Cu_2Cy_6Fe), which is insoluble in dilute acids, but is decomposed by potash. In very dilute solutions the reagent produces only a reddish coloration.

11. *Metallic iron*, when brought into contact with concentrated solutions of copper, is almost immediately coated with metallic copper. Very dilute solutions produce this coating only after some time. A little free acid accelerates the reaction.

12. If to a solution of copper in a platinum crucible lid a little hydrochloric acid is added, and then a piece of zinc, the platinum will soon become coated with copper; this coating is clearly visible even in the case of very dilute solutions.

13. If a piece of iron wire is inserted into a spiral formed

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* In the presence of non-volatile organic acids, the salts of copper are not precipitated by potash or carbonate of sodium; the solutions produced are deep blue. In solutions containing sugar, or a similar organic substance, potash produces a precipitate soluble in excess; carbonate of sodium, however, gives a permanent precipitate.

from a rather stout platinum wire, and the whole is then placed in a slightly acidified solution of copper, the platinum wire will after some time be found to be coated with copper.

Cadmium.

WET REACTIONS.

1. *Hydrosulphuric acid* produces a bright yellow precipitate of sulphide of cadmium (CdS), which is insoluble in dilute acids, alkalies, alkaline sulphides, and cyanide of potassium. It is readily dissolved by boiling nitric acid, by boiling hydrochloric acid, and by boiling sulphuric acid.

2. *Sulphide of ammonium* produces the same precipitate.

3. *Potash* produces a white precipitate of hydrate of cadmium (CdH_2O_2), which is insoluble in an excess of the precipitant.

4. *Ammonia* produces the same precipitate. It is readily soluble in an excess of this reagent.

5. *Carbonate of sodium* produces a white precipitate of carbonate of cadmium ($CdCO_3$), which is insoluble in excess of the precipitant. Salts of ammonium interfere with the precipitation notably; ammonia prevents it. The precipitate dissolves readily in cyanide of potassium. From dilute solutions the precipitate separates only after some time. Warming assists the separation.

6. *Carbonate of ammonium* produces the same precipitate, also insoluble in excess of the precipitant.

GROUP II.—DIVISION II.

TIN. ANTIMONY. ARSENIC.

Tin.

DRY REACTION.

1. Compounds of tin fused with carbonate of sodium and cyanide of potassium on *charcoal* give malleable globules of metallic tin.

WET REACTIONS.

Dyad Tin.

2. *Hydrosulphuric acid* produces in acid solutions a dark

brown precipitate of hydrated stannous sulphide (SnS), which is insoluble, or nearly so, in colorless sulphide of ammonium, but dissolves readily in the yellow sulphide. Acids precipitate from this solution yellow stannic sulphide, mixed with sulphur. Stannous sulphide dissolves also in potash; acids precipitate from this solution the stannous sulphide unchanged. Boiling hydrochloric acid dissolves it; boiling nitic acid converts it into insoluble metastannic acid (SnH_4O_4).

3. *Potash* produces a white bulky precipitate of stannous hydrate (SnH_2O_2) which redissolves readily in excess of the precipitant. If the solution is briskly evaporated a compound of stannic oxide and potash is formed, which remains in solution, while metallic tin precipitates; but upon evaporating it slowly, crystalline anhydrous stannous oxide separates.

4. *Ammonia* produces the same precipitate, insoluble in excess of the precipitant.

5. *Carbonate of ammonium* produces the same precipitate insoluble in excess of the precipitant.

6. *Mercuric chloride,* added in excess, produces a white precipitate of mercurous chloride. See p. 32, 7, top.

7. *Zinc* precipitates from solutions containing excess of hydrochloric acid, metallic tin in the form of grey laminæ or a spongy mass. If the experiment is conducted in a platinum dish the latter is not colored black.

Tetrad Tin.

Note.—There are two series of stannic compounds, the stannic and the metastannic. The stannic hydrate precipitated by alkalies from stannic chloride dissolves readily in hydrochloric acid, but the stannic acid obtained by the action of nitric acid on tin does not dissolve in hydrochloric acid. If, however, this stannic acid is boiled with hydrochloric acid for a short time, it takes up acid, and on pouring off the supernatant liquid and adding water to the residue, a solution is obtained. The aqueous solution of the ordinary stannic chloride is not precipitated by strong hydrochloric acid, whilst the metastannic chloride is precipitated unchanged from its aqueous solution by this acid. The solution of the ordinary stannic chloride is not turned yellow by stannous chloride, but if it contains metastannic chloride it is turned yellow (LÖWEN-

THAL). The dilute solutions of the stannic chlorides when boiled, yield stannic hydrate or metastannic acid, as the case may be.

1. *Hydrosulphuric acid* throws down from acid solutions, particularly upon heating, a white flocculent precipitate if the stannic solution is in excess, a faintly yellow precipitate if the hydrosulphuric acid is in excess. The former precipitate may be safely assumed to be, in the case of stannic chloride, a mixture of stannic chloride and sulphide; the latter precipitate consists of hydrated stannic sulphide (SnS_2). The stannic sulphide dissolves readily in potash, alkaline sulphides, boiling strong hydrochloric acid, and aqua regia. It dissolves with some difficulty in ammonia, and is nearly insoluble in carbonate of ammonium, and in acid sulphite of potassium. It dissolves readily in sulphide of ammonium; from this solution acids precipitate the stannic sulphide unaltered. Concentrated nitric acid converts it into insoluble metastannic acid (SnH_4O_4). Upon deflagrating stannic sulphide with nitrate and carbonate of sodium, stannic oxide and sulphate of sodium are formed.

2. *Potash* produces a white precipitate, which, according to the nature of the solution, consists of stannic hydrate or metastannic acid. Both dissolve readily in excess of the precipitant.

3. *Ammonia* produces the same precipitate.

4. *Carbonate of ammonium* produces the same precipitate.

5. *Sulphate of sodium*, or nitrate of ammonium (in fact, most neutral salts of the alkali metals), when added in excess, throw down from solutions of both varieties, provided they are not too acid, the whole of the tin as stannic hydrate or metastannic acid. Heating promotes the precipitation.

6. *Zinc* precipitates from solutions containing excess of hydrochloric acid, metallic tin in the form of grey laminæ or of a spongy mass. If the experiment is conducted in a platinum vessel the latter is not colored black.

Antimony.

DRY REACTION.

1. Compounds of antimony mixed with carbonate of sodium and exposed on *charcoal* to the reducing blowpipe-flame, yield brittle globules of metallic antimony.

WET REACTIONS.

2. *Hydrosulphuric acid* added to acid solutions produces an orange-red precipitate of antimonious sulphide (Sb_2S_3). The precipitate is readily dissolved by potash and by alkaline sulphides, especially if the latter contain an excess of sulphur. It is but sparingly soluble in ammonia, and, if free from antimonic sulphide, almost insoluble in acid carbonate of ammonium. It is insoluble in dilute acids and in acid sulphite of potassium. Boiling strong hydrochloric acid dissolves it. By heating in the air it is converted into a mixture of binoxide of antimony and antimonious sulphide. By deflagration with nitrate and carbonate of sodium it gives sulphate of sodium and antimoniate of sodium ($Na_2Sb_2O_6$). On fusing antimonious sulphide with cyanide of potassium metallic antimony is obtained. If the operation is conducted in an ignition-tube, or in a stream of carbonic acid, no sublimate of antimony is produced. But if a mixture of sulphide of antimony with cyanide of potassium and carbonate of sodium is heated in a glass tube in a stream of hydrogen, a mirror of antimony is deposited in the tube immediately in front of the spot occupied by the mixture.

3. *Potash** throws down a white bulky precipitate of antimonious oxide (Sb_2O_3), which redissolves pretty readily in excess of the precipitant.

4. *Ammonia** produces the same precipitate, which is almost insoluble in excess of the precipitant.

5. *Carbonate of ammonium** produces the same precipitate.

6. *Zinc* precipitates metallic antimony as a black powder, if free nitric acid is absent. If a few drops of a solution of antimony are put into a platinum crucible lid, with a little hydrochloric acid and a piece of zinc, hydrogen containing antimonetted hydrogen is evolved and antimony separates, staining the platinum brown or black, even in the case of very dilute solutions. This reaction is exceedingly delicate and characteristic. Cold hydrochloric acid fails to remove the stain; heating with nitric acid removes it immediately.

7. If a minute quantity of antimony in solution is intro-

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* These reagents precipitate tartar-emetic very imperfectly.

duced into an apparatus evolving hydrogen, and fitted with a jet, part of the antimony will be precipitated as metal and remain in the apparatus, while part will escape with the hydrogen in the form of antimonetted hydrogen (SbH_3). If the escaping gas is lighted, and a porcelain dish is held in the flame, a black stain or mirror of antimony will be deposited on the dish. If the jet tube is bent at right angles and heated at some little distance from the end, a mirror will be formed in the tube on both sides of the heated part.

Arsenic gives a similar mirror when treated as above. It is therefore necessary to be able to distinguish between the two. The mirror on the porcelain is best tested with a drop of chloride of soda—this does not dissolve the antimony mirror, while it would at once dissolve an arsenic mirror. The mirror in the tube gives the following reactions:—When heated in the tube in the current of hydrogen it only volatilizes at a high temperature, and the hydrogen then escaping is devoid of garlic odor; when the hydrogen is lighted it only deposits spots on porcelain if the current of gas is strong; and the mirror before volatilizing fuses to shining globules distinctly visible with a lens. But the most satisfactory way of proving the identity of the antimony mirror in the tube, is by conducting a very slow current of dry sulphuretted hydrogen through the tube and heating the mirror, moving the lamp in the opposite direction to that of the current. The antimony mirror is by this means converted into antimonious sulphide, which appears of a more or less reddish-yellow color, and almost black when in thick layers. If a gentle current of dry hydrochloric acid is now transmitted through the tube, the sulphide of antimony, if present in thin layers only, disappears immediately; if the incrustation is somewhat thick, a short time is required to dissipate it. The reason of this is that sulphide of antimony is readily decomposed by hydrochloric acid, and the terchloride of antimony formed is very volatile in a stream of hydrochloric acid. If the gas, as it passes out of the tube, is conducted into water, the antimony is dissolved and may be detected with hydrosulphuric acid. By this combination of reactions, antimony may be distinguished with certainty from all other metals.

If hydrogen containing antimonetted hydrogen is slowly passed into solution of nitrate of silver, almost the whole of the antimony is precipitated in the form of $SbAg_3$.

$$SbH_3+3AgNO_3=SbAg_3+3HNO_3.$$

8. *Water* throws down from solutions of antimony a precipitate of basic salt. Thus, on adding water to solution of chloride of antimony in hydrochloric acid, a white bulky precipitate of basic antimonious chloride ($2SbCl_3 . 5Sb_2O_3$) is produced, which, after some time, becomes heavy and crystalline. Tartaric acid dissolves this precipitate readily (here antimony differs from bismuth), and therefore prevents its formation if mixed with the solution before the addition of water.

9. If a solution of antimonious oxide in potash is mixed with nitrate of silver, a deep black precipitate of suboxide of silver (Ag_4O) forms along with the greyish-brown precipitate of oxide of silver. Upon now adding ammonia in excess the oxide is redissolved, while the suboxide is left. This reaction is very delicate. It is thus explained:—

$$Sb_2O_3+4Ag_2O=Sb_2O_5+2Ag_4O.$$

10. When antimonious oxide is boiled with hydrochloric acid and iodide of potassium, no iodine is liberated.

Antimonic Acid.

1. *Hydrosulphuric acid* produces in acid solutions a precipitate of antimonic sulphide (Sb_2S_5) mixed with antimonious sulphide and sulphur. The precipitate dissolves readily when heated with potash or ammonia. It dissolves in boiling strong hydrochloric acid with evolution of sulphuretted hydrogen and separation of sulphur. It is only very sparingly soluble in cold acid carbonate of ammonium.

2. With *zinc* and in a *hydrogen apparatus*, solutions of antimonic acid show the same reactions as ordinary solutions of antimony.

3. If a solution of antimonic acid in potash is mixed with *nitrate of silver* and then with ammonia, no suboxide of silver is left behind.

4. If antimonic acid is boiled with hydrochloric acid and iodide of potassium, iodine is liberated, and colors the solution brown.

Arsenic.

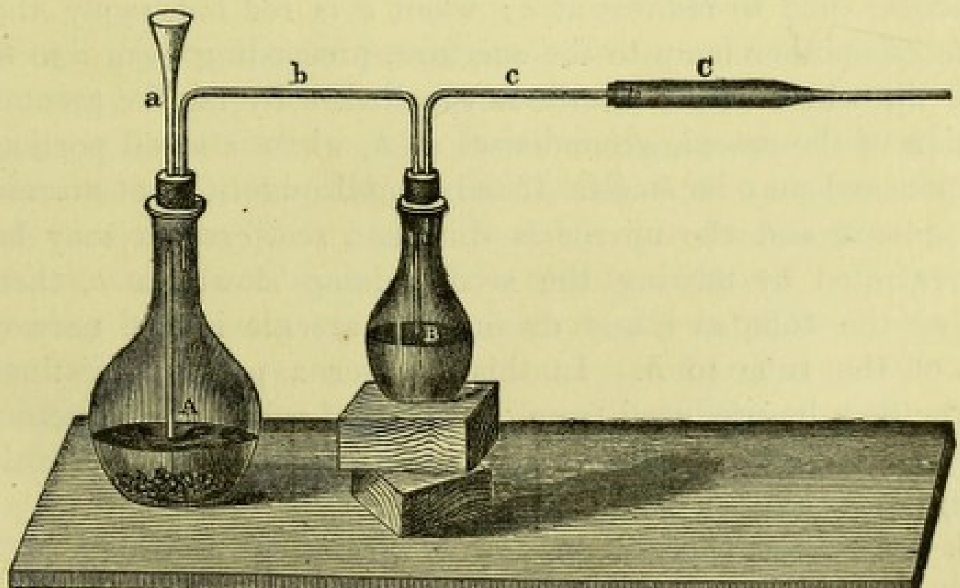
DRY REACTIONS.

1. Compounds of arsenic heated with carbonate of sodium on *charcoal* in the inner blowpipe flame, give a highly characteristic garlic odor. This reaction is delicate.

2. Compounds of arsenic heated with *carbonate of sodium and cyanide of potassium* in an *ignition-tube* yield a sublimate of metallic arsenic in the form of a mirror. It is best to blow the sealed end of the ignition-tube into a bulb, and to proceed as follows:—Introduce the perfectly dry substance into the bulb and cover it with five times its bulk of the perfectly dry reducing mixture. The whole quantity must not occupy more than half the bulb, otherwise when the mixture fuses it may ascend into the tube. Now heat the bulb gently; should some water escape, wipe the inside of the tube perfectly dry with a twisted piece of filter-paper. (It is necessary that the mixture and tube should be perfectly free from moisture.) Apply a strong heat to the bulb and continue this for some time, as the arsenic does not always sublime completely at once. This method is simple, neat, and delicate; it is especially adapted for the production of a mirror from the sulphide of arsenic.

The delicacy of this reaction is heightened by heating the mixture in a current of dry carbonic acid. The disposition of the apparatus is shown in the figure; *A* contains marble and

Fig. 3.



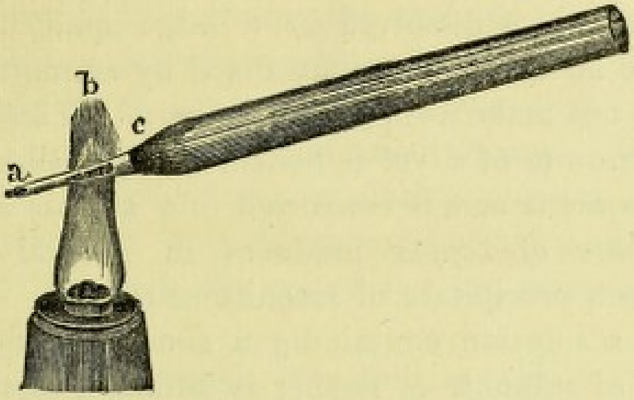
water, and is supplied with hydrochloric acid through the funnel tube. This flask may be conveniently replaced by an apparatus for giving a constant supply of the gas, which can be regulated at will. *B* contains sulphuric acid; *C* is 30 cm. long; it is made of a piece of hard glass tube, rather stout, and of about 7 mm. bore; fig. 4 is an enlarged drawing of it. When the apparatus is full of carbonic acid, rub the dry sulphide or other compound of arsenic in a warm mortar with about ten parts of a well-dried mixture consisting of three parts of carbonate of sodium and one part of cyanide of potassium. Put the powder upon a narrow slip of thick paper bent into the shape of a gutter, and push this into the reduction tube down to *e*; turn the tube now half way round its axis, which will cause the mixture to drop into the tube between *e* and *d*, every other part remaining perfectly clean. Connect the tube with the rest of the apparatus, and pass a gentle current of gas. Heat the reduction tube in its whole length very gently, until the mixture is perfectly dry. When every trace of moisture has been expelled from the tube, and the current is passing through the sulphuric acid at the rate of a bubble a second, heat the reduction tube to redness at *c*; when *c* is red hot, apply the flame of another lamp to the mixture, proceeding from *d* to *e*, until the whole of the arsenic is expelled. By far the greater portion of the arsenic recondenses at *h*, while a small portion escapes, and may be smelt. If only small quantities of arsenic are present, and the mirror is thin and scattered, it may be concentrated by moving the second lamp slowly to *c*, then sealing the tube at *i*, and driving the arsenic in the narrow part of the tube to *h*. In this manner a perfectly distinct mirror may be produced from ·2 mgrm. of sulphide of arsenic. No mirror is obtained by this process from sulphide of antimony, or any other compound of antimony.

FIG. 4.

3. *The best way of identifying arsenious oxide* (As_2O_3) when unmixed with other bodies consists in the two following reactions:—First, heat in an ignition tube; the arsenious oxide will sublime into the form of small sparkling octa-

hedrons and tetrahedrons. Secondly, introduce a fragment into the pointed end (*a*) of an ignition-tube drawn out as

FIG. 5.



shown in the figure, then insert a piece of recently ignited charcoal (*b*), and heat to redness first the charcoal, afterwards the arsenious oxide. A mirror of arsenic will form at *c*. If the tube is now cut between *b* and *c*, and then heated in an inclined position, with *c* upwards, the mirror will volatilize, emitting the characteristic odor. No other substance would give sparkling crystals when heated in an ignition-tube, and an arsenical mirror when heated with charcoal as described.

WET REACTIONS.

Arsenious Acid.

4. *Hydrosulphuric acid* produces in acidified solutions a bright yellow precipitate of arsenious sulphide (As_2S_3). The precipitate forms at once. It is readily dissolved by alkalies, alkaline carbonates and sulphides, and acid carbonates of the alkali metals. It is nearly insoluble in hydrochloric acid, even when strong and boiling. It is readily soluble in boiling nitric acid. If recently precipitated arsenious sulphide is digested with sulphurous acid and acid sulphite of potassium, the precipitate is dissolved ; upon heating the solution to boiling the fluid turns turbid, owing to the separation of sulphur, which upon continued boiling is for the greater part redissolved. The fluid contains, after expulsion of the sulphurous acid, arsenite and hyposulphite of potassium. The deflagration of arsenious sulphide with carbonate and nitrate of sodium gives rise to the formation of arseniate and sulphate of sodium.

5. *Nitrate of silver* produces in neutral solutions a yellow precipitate of arsenite of silver (Ag_3AsO_3). The precipitate is readily soluble in nitric acid and in ammonia. It is not insoluble in nitrate of ammonium; hence if a small quantity of the precipitate is dissolved in a large quantity of nitric acid, and the latter is then neutralized by ammonia, the precipitate does not make its appearance again. If an ammoniacal solution of arsenite of silver is boiled, metallic silver separates, while the arsenious acid is converted into arsenic acid.

6. *Sulphate of copper* produces in neutral solutions a yellowish-green precipitate of arsenite of copper.

7. If to a solution containing a good excess of potash, a drop or two of sulphate of copper is added, a clear blue fluid is obtained, which upon boiling deposits a red precipitate of cuprous oxide (Cu_2O), leaving arseniate of potassium in solution. This reaction is exceedingly delicate provided not too much sulphate of copper is used. Even should the red precipitate be so trifling as to escape detection by transmitted light, yet it will always be discernible upon looking in at the top of the test-tube. This reaction is valuable for the detection of arsenious acid in presence of arsenic acid; but it must be noticed that grape sugar and other organic substances show a similar deportment.

8. *Reinsch's Test.*—If a solution mixed with hydrochloric acid is heated with a clean piece of copper-foil or wire, an iron grey film of Cu_5As_2 is formed on the copper, even in highly dilute solutions; when this film increases in thickness, it peels off in black scales. If the coated copper is washed free from acid, and then heated with ammonia, the film peels off from the copper, and separates in the form of minute spangles. If the substance, either simply dried, or oxidized by ignition in a current of air (which is attended with the escape of some arsenious oxide), is heated in a current of hydrogen, there escapes relatively but little arsenic, alloys richer in copper being left behind.

This reaction is not decisive unless arsenic is found in the film, as antimony and other metals will precipitate upon copper under similar circumstances.

9. *Marsh's Test.*—If a solution is mixed with zinc and dilute sulphuric acid, arsenetted hydrogen (AsH_3) is formed in the same manner as antimonetted hydrogen is formed from

compounds of antimony. This reaction affords us a most delicate test for arsenic.

The operation is conducted in a bottle supplied with a funnel-tube and a jet-tube; the latter should be bent at right angles, and a wide tube containing chloride of calcium (to dry the gas) should be intercepted between it and the bottle. The jet-tube should be of hard glass, free from lead; it should be rather stout, and have a bore of about 7 mm. The operation is commenced by evolving a moderate current of hydrogen from granulated zinc and dilute sulphuric acid.* When the evolution of hydrogen has proceeded for some time, the gas should be lighted.

We must now first make sure that our zinc and sulphuric acid are free from arsenic. This is done by depressing a porcelain dish upon the flame, so as to make it spread over the surface; if the hydrogen contains arsenetted hydrogen, brownish, or brownish-black, stains will appear on the porcelain; the non-appearance of such stains may be taken as a proof that the materials are free from arsenic. In very delicate experiments, however, additional evidence is required to prove the absence of the slightest trace of arsenic in the reagents; for this purpose, part of the jet-tube is heated to redness, and kept for some time in that state: if no arsenical coating makes its appearance, the hydrogen may be considered pure.

The operation is proceeded with by pouring the fluid to be tested through the funnel-tube into the bottle, and afterwards a little water to rinse out the tube. Only a very little of the fluid should be poured in at first, as when the quantity of arsenic present is considerable, and much fluid is poured into the bottle, the evolution of gas often proceeds with such violence as to stop the further progress of the experiment. If a porcelain dish is now held in the flame, metallic arsenic condenses upon it, in the same manner as antimony. The arsenical mirror is not quite similar to the antimony mirror in appearance; it is, however, readily distinguished from the latter by its solubility in chloride of soda.

If a part of the jet-tube is heated, a brilliant arsenical

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* If pure zinc is used, a difficulty may be found in obtaining an action; in that case a few drops of chloride of platinum should be added.

mirror is produced in the tube. On heating this mirror in the current of hydrogen, it is readily driven away without previously melting, while at the same time the escaping (unlighted) gas possesses the characteristic arsenical odor. If the gas is lighted while the mirror in the tube is being heated, the flame will, even with a very slight current of gas, deposit arsenical stains on a porcelain dish.

The reactions just described are amply sufficient to enable us to distinguish between arsenical and antimonial mirrors; but they are often inadequate to the positive detection of arsenic in the presence of antimony. In cases of this kind, the following process will serve to set at rest all possible doubt as to the presence or absence of arsenic:—

While the gas is passing, heat the long jet-tube to redness in several parts, to produce several distinct mirrors; then transmit through the tube a very slow current of dry sulphuretted hydrogen, and heat the mirrors, moving the lamp in the opposite direction to that of the current. If arsenic alone is present, yellow sulphide of arsenic is formed in the tube; if antimony alone is present, an orange-red or black sulphide of antimony is produced; and if the mirror consisted of both metals, the two sulphides appear side by side, the sulphide of arsenic, as the more volatile, lying invariably in front of the sulphide of antimony. If you now transmit through the tube dry hydrochloric acid, without applying heat, no alteration will take place if sulphide of arsenic alone is present, even though the gas be transmitted for a considerable time. If sulphide of antimony alone is present, this will entirely disappear; and if both sulphides are present, the sulphide of antimony will immediately volatilize, while the yellow sulphide of arsenic will remain. If a small quantity of ammonia is now drawn up into the tube, the sulphide of arsenic is dissolved, and may then be readily distinguished from any sulphur which may have separated. My personal experience has convinced me of the infallibility of these combined tests for the detection of arsenic.

If hydrogen containing arsenetted hydrogen is passed slowly into nitrate of silver, the arsenic is retained in the form of arsenious acid, while metallic silver is precipitated.

$$AsH_3 + 6AgNO_3 + 3H_2O = Ag_6 + H_3AsO_3 + 6HNO_3.$$

Arsenic Acid.

1. *Hydrosulphuric acid* causes in acid solutions, first reduction of the arsenic acid to arsenious acid, with separation of sulphur, then precipitation of arsenious sulphide. This process continues until the whole of the arsenic is thrown down, as As_2S_3 mixed with S_2. The action never takes place immediately, and in dilute solutions it frequently requires a considerable time (twelve or twenty-four hours for instance). Heating to about 70° greatly accelerates the reaction.

In precipitating arsenic acid solutions with hydrosulphuric acid, it is often most convenient to reduce the arsenic acid to arsenious acid previously. The hydrosulphuric acid will then precipitate the arsenic as arsenious sulphide immediately. The reduction is effected by heating with sulphurous acid which is converted into sulphuric acid in the action.

2. *Nitrate of silver* produces in neutral solutions a highly characteristic reddish-brown precipitate of arseniate of silver (Ag_3AsO_4), which is readily soluble in dilute nitric acid and in ammonia. It is slightly soluble in nitrate of ammonium; accordingly, if a little of it is dissolved in a large proportion of nitric acid, neutralization with ammonia often fails to reproduce the precipitate. When the solution of the precipitate in ammonia is boiled, no metallic silver separates, as is the case with arsenite of silver.

3. *Sulphate of copper* produces in neutral solutions a greenish-blue precipitate of arseniate of copper ($CuHAsO_4$).

4. A mixture of *sulphate of magnesium*, chloride of ammonium, and ammonia, produces a crystalline precipitate of arseniate of ammonium and magnesium ($MgNH_4AsO_4 + 6$ aq). In dilute solutions the precipitate takes some time to form.

This precipitate may be distinguished from the corresponding phosphate by the following reactions :—(a) If a small quantity of the precipitate is dissolved in a watch glass in a drop of nitric acid, a little nitrate of silver added, and the solution touched with a glass rod that has been dipped in ammonia, a brownish-red precipitate of arseniate of silver is formed. (b) If the arseniate of magnesium and ammonium is dissolved in hydrochloric acid, and hydrosulphuric acid is passed into the solution which is kept warm, a yellow precipitate will be formed.

5. If a dilute solution is mixed with some hydrochloric acid and heated with a clean slip of copper, the metal remains perfectly unchanged; but if to one volume of the solution two volumes of strong hydrochloric are added, a grey film is deposited on the copper, as with arsenious acid. Under these circumstances the reaction is as delicate with arsenic acid as with arsenious acid.

6. With *zinc and sulphuric acid* arsenic solutions give the same reactions as arsenious solutions.

GROUP III.

NICKEL. COBALT. IRON. MANGANESE. ZINC. CHROMIUM. ALUMINIUM.

Nickel.

DRY REACTION.

1. Compounds of nickel heated in the outer blowpipe flame with the *borax bead* give a reddish-brown color when cold.

WET REACTIONS.

2. *Sulphide of ammonium* produces a black precipitate of hydrated sulphide of nickel (NiS), which is not altogether insoluble in excess of the precipitant, especially if it contains free ammonia; if the mixture is filtered, therefore, the filtrate usually exhibits a brown color. Chloride of ammonium promotes the precipitation, and acetate of ammonium is still more favorable. Sulphide of nickel dissolves scarcely at all in acetic acid, with great difficulty in hydrochloric acid, but readily in aqua regia upon application of heat.

3. *Potash*[*] produces a light green precipitate of hydrate of nickel (NiH_2O_2) which is insoluble in an excess of the precipitant, and unalterable in the air and by boiling. Carbonate of ammonium dissolves the precipitate, when filtered and washed, to a greenish-blue fluid, from which potash reprecipitates the nickel as apple-green hydrate.

4. *Ammonia* added in small quantity to a neutral solution,

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[*] The presence of non-volatile organic acids and sugar, interferes with the precipitation by potash.

produces a trifling greenish turbidity, which at once redissolves to a blue fluid on further addition of the reagent. Potash precipitates from this solution hydrate of nickel. Presence of salts of ammonium prevents even any precipitate being produced by ammonia.

5. *Ferrocyanide of potassium* precipitates ferrocyanide of nickel (Ni_2Cy_6Fe), as a greenish-white body, insoluble in hydrochloric acid.

6. *Ferricyanide of potassium* precipitates brownish-yellow ferricyanide of nickel [$Ni_3(Cy_6Fe)_2$], which is insoluble in hydrochloric acid.

7. *Cyanide of potassium* produces a yellowish-green precipitate of cyanide of nickel ($NiCy_2$), which dissolves readily in excess of the precipitant as cyanide of nickel and potassium (K_2NiCy_4); the solution is brownish-yellow. If hydrochloric acid is added to this solution, the cyanide of nickel is re-precipitated. From very dilute solutions the cyanide of nickel separates only after some time; it is very difficultly soluble in an excess of the hydrochloric acid in the cold, but more readily soluble on boiling.

The solution of cyanide of nickel and potassium is not altered by boiling with excess of hydrocyanic acid. It gives a black precipitate of sesquioxide of nickel when boiled with chloride of soda.

8. *Carbonate of barium* does not precipitate nickel upon digestion in the cold.

9. *Nitrite of potassium* used in conjunction with acetic acid fails to precipitate even concentrated solutions of nickel.

If the solution, however, contains calcium, barium, or strontium, and it is not too dilute, a yellow crystalline precipitate of nitrite of nickel and calcium, &c., is formed, which is difficultly soluble in cold water, and more readily soluble in hot water to a yellow fluid.

Cobalt.

DRY REACTION.

1. Compounds of cobalt heated in either the inner or outer blowpipe flame with *borax* give a magnificent blue bead. This test is both delicate and characteristic.

WET REACTIONS.

2. *Sulphide of ammonium* precipitates black hydrated sulphide of cobalt (CoS). The precipitation is promoted by chloride of ammonium. Sulphide of cobalt is insoluble in alkalies and sulphide of ammonium, scarcely soluble in acetic acid, very difficultly soluble in hydrochloric acid, but readily soluble in aqua regia, upon application of heat.

3. *Potash** produces a blue precipitate of basic salt which is insoluble in excess of the precipitant, and turns green upon exposure to the air, owing to the absorption of oxygen. Upon boiling it is converted into pale red hydrate of cobalt (CoH_2O_2), which contains alkali, and generally appears rather discolored from an admixture of sesquioxide. If before boiling alcohol is added, the precipitate is rapidly converted into dark brown hydrated sesquioxide. Carbonate of ammonium dissolves the basic salt (or the hydrate of cobalt) after it has been washed completely, forming an intensely colored violet-red fluid, in which a blue precipitate is produced by potash, while the fluid remains violet.

4. *Ammonia* produces in neutral solutions a precipitate of basic salt, which readily redissolves in excess of the precipitant to a reddish fluid which turns brownish-red on exposure to the air, and from which potash throws down part of the cobalt as a basic salt. Presence of salts of ammonium prevents even any precipitate being produced by ammonia.

5. *Ferrocyanide of potassium* precipitates green ferrocyanide of cobalt (Co_2Cy_6Fe), which is insoluble in hydrochloric acid.

6. *Ferricyanide of potassium* precipitates brownish-red ferricyanide of cobalt [$Co_3(Cy_6Fe)_2$], which is insoluble in hydrochloric acid.

7. *Cyanide of potassium* produces a brownish-white precipitate of cyanide of cobalt ($CoCy_2$), which dissolves readily in excess of the precipitant as cyanide of cobalt and potassium (K_2CoCy_4). Acids reprecipitate from this solution the cyanide of cobalt.

If this solution of cyanide of cobalt and potassium is boiled with more cyanide of potassium, in presence of hydrocyanic

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* The presence of non-volatile organic acids or sugar interferes with the precipitation of cobalt by potash.

acid (produced by the addition of a drop or two of hydrochloric acid), cobalticyanide of potassium (K_3Cy_6Co) is formed, thus—

$$K_2CoCy_4 + KCy + HCy = K_3Cy_6Co + H$$

The solution of cobalticyanide of potassium is not precipitated by acids or by boiling with chloride of soda.

8. *Carbonate of barium* fails to precipitate cobalt, upon digestion in the cold.

9. If *nitrite of potassium* is added in not too small proportion to a solution of cobalt, then acetic acid to strongly acid reaction, and the mixture is put in a moderately warm place, all the cobalt separates in the form of a crystalline precipitate of a fine yellow color. The separation takes some time in the case of dilute solutions. The precipitate has the following composition :—$Co_2O_3 . 3K_2O . 5N_2O_3 . 2H_2O$. Its formation may be thus explained :—

$$2CoCl_2 + 12KNO_2 + 2H\bar{A} + H_2O = Co_2O_3 . 3K_2O . 5N_2O_3 . 2H_2O$$
$$+ 4KCl + 2K\bar{A} + 2NO.$$

The precipitate is very perceptibly soluble in water, only slightly soluble in concentrated solutions of salts of potassium and in alcohol, and insoluble in presence of nitrite of potassium. When boiled with water, it dissolves, but not copiously, to a red fluid, which remains clear upon cooling, and from which potash throws down hydrate of cobalt.

Iron.

DRY REACTION.

1. The *borax bead* is colored by compounds of iron. Heated in the outer blowpipe flame, the bead when cold varies from colorless to dark yellow. Heated in the inner flame, the bead when cold is bottle green.

WET REACTIONS.

Dyad Iron.

2. *Sulphide of ammonium* precipitates black hydrated ferrous sulphide (FeS), which is insoluble in alkalies and sulphides of the alkali metals, but dissolves readily in hydrochloric and nitric acids. The precipitate turns reddish-brown in the air by oxidation. To highly dilute solutions sulphide of ammonium imparts a green color; after some time the iron

falls down as black sulphide. Chloride of ammonium promotes the precipitation most materially.

3. *Potash** produces a precipitate of ferrous hydrate (FeH_2O_2) which is at first almost white; it soon becomes dirty green, and ultimately reddish-brown, from absorption of oxygen. Presence of ammonium salts prevents the precipitation partly.

4. *Ammonia** produces the same precipitate. Presence of salts of ammonium prevents the precipitation altogether.

5. *Ferrocyanide of potassium* produces a bluish-white precipitate of ferrocyanide of potassium and iron [$K_2Fe_3(Cy_6Fe)_2$]. Its formation may be explained thus:—

$$3FeSO_4+2K_4Cy_6Fe=K_2Fe_3(Cy_6Fe)_2+3K_2SO_4$$

The precipitate speedily becomes blue from absorption of oxygen. Chlorine converts it immediately into Prussian blue [$Fe_4(Cy_6Fe)_3$], thus:—

$$3K_2Fe_3(Cy_6Fe)_2+Cl_9=2Fe_4(Cy_6Fe)_3+6KCl+FeCl_3$$

6. *Ferricyanide of potassium* produces a fine blue precipitate of ferrous ferricyanide [$Fe_3(Cy_6Fe)_2$]. The color of the precipitate is the same as that of Prussian blue. It is insoluble in hydrochloric acid, but is readily decomposed by potash. In highly dilute solutions the reagent produces simply a deep bluish-green coloration.

7. *Sulphocyanide of potassium* does not alter ferrous solutions.

8. *Carbonate of barium* does not precipitate ferrous solution in the cold.

Triad Iron.

1. *Hydrosulphuric acid* produces in acid solutions a milky-white turbidity proceeding from separated sulphur; the ferric salt being reduced to a ferrous salt, thus:—

$$2FeCl_3+H_2S=2FeCl_2+2HCl+S$$

2. *Sulphide of ammonium* produces a black precipitate, consisting of hydrated ferrous sulphide (FeS) mixed with sulphur, thus:—

$$2FeCl_3+3(NH_4)_2S=2FeS+S+6NH_4Cl.$$

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* Non-volatile organic acids, sugar, &c., interfere with the precipitation of ferrous salts by alkalies.

For the properties of the precipitate see p. 51, 2.

3. *Potash** produces a bulky, reddish-brown precipitate of ferric hydrate (FeH_3O_3), which is insoluble in excess of the precipitant and in salts of ammonium.

4. *Ammonia** produces the same precipitate, insoluble in excess of the precipitant.

5. *Ferrocyanide of potassium* produces, even in very dilute solutions, a fine blue precipitate of Prussian blue or ferric ferrocyanide [$Fe_4(Cy_6Fe)_3$]. This precipitate is insoluble in hydrochloric acid, but is decomposed by potash, with separation of ferric hydrate.

6. *Ferricyanide of potassium* deepens the color of ferric solutions to reddish-brown, but it produces no precipitate.

7. *Sulphocyanide of potassium*† imparts to acid solutions an intense blood-red color, arising from the formation of soluble ferric sulphocyanide. This test is the most delicate of all; it will indicate the presence of a ferric salt when all other tests fail. The red coloration may in such cases be detected more distinctly by resting the tube upon a piece of white paper, and looking through it from the top. The sensitiveness of the reaction may be increased by adding hydrochloric acid and then ether, and shaking. The ferric sulphocyanide dissolves in the ether, and the color is concentrated.

8. *Carbonate of barium* (even in the cold) precipitates the iron as ferric hydrate, mixed with basic salt.

Manganese.

DRY REACTIONS.

1. The *borax bead*, when heated with compounds of manganese in the outer blowpipe flame, acquires when cold an amethyst-red color. The bead loses its color in the inner flame.

2. If a *bead of carbonate of sodium* is made in the loop of a platinum wire, touched with a finely-divided compound

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* Non-volatile organic acids and sugar prevent the precipitation of ferric salts by alkalies.

† In ferric solutions which have been mixed with acetate of sodium (and are more or less red from the formation of ferric acetate), no ferric sulphocyanide will be formed until a considerable quantity of hydrochloric acid has been added. The same remark applies to solutions containing an oxalate.

of manganese, held in the flame, then dipped into powdered nitrate of potassium and heated again in the blowpipe flame, a green bead will be obtained, which becomes bluish on cooling. This reaction depends upon the formation of manganate of sodium (Na_2MnO_4). It enables us to detect the smallest traces of manganese.

WET REACTIONS.

3. *Sulphide of ammonium* throws down hydrated sulphide of manganese (MnS) in the form of a light flesh-colored precipitate. If the solution is very dilute, the color of the precipitate may appear yellowish white. The precipitate acquires a dark brown color in the air. It is insoluble in sulphide of ammonium and in alkalies; but readily soluble in hydrochloric, nitric, and acetic acids. The separation of the precipitate is materially promoted by chloride of ammonium. From very dilute solutions the precipitate separates only after standing some time in a warm place. Solutions containing much free ammonia should be nearly neutralized with hydrochloric acid before precipitation with sulphide of ammonium. If ammonia and sulphide of ammonium are present in considerable excess, the flesh-colored hydrated precipitate often changes in the cold to the green anhydrous sulphide—this alteration occurs rapidly on boiling. Chloride of ammonium interferes with the conversion.

4. *Potash** produces a whitish precipitate of manganous hydrate (MnH_2O_2), which upon exposure to the air soon acquires a brown color from absorption of oxygen.

5. *Ammonia** produces the same precipitate. The precipitation is prevented by the presence of chloride of ammonium.

6. *Ferrocyanide of potassium* throws down manganous ferrocyanide (Mn_2Cy_6Fe). It is reddish-white and soluble in hydrochloric acid.

7. *Ferricyanide of potassium* precipitates manganous ferricyanide [$Mn_3(Cy_6Fe)_2$] as a brown body, insoluble in hydrochloric acid and ammonia.

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* Non-volatile organic acids may prevent the precipitation by alkalies (or alkaline carbonates). Sugar interferes with the precipitation by alkalies, but it does not interfere with the precipitation by alkaline carbonates.

8. *Carbonate of barium* fails to throw down any manganese on digestion in the cold.

Zinc.

WET REACTIONS.

1. *Sulphide of ammonium* produces a white precipitate of hydrated sulphide of zinc (ZnS). Chloride of ammonium greatly promotes the separation of the precipitate. From very dilute solutions the precipitate only separates after long standing. The precipitate is insoluble in excess of sulphide of ammonium, and also in potash and ammonia. It dissolves readily in hydrochloric acid, nitric acid, and dilute sulphuric acid. It is insoluble in acetic acid.

2. *Potash** throws down hydrate of zinc (ZnH_2O_2), in the form of a white gelatinous precipitate, which is readily and completely redissolved by an excess of the precipitant. Upon boiling this solution it remains unaltered, if concentrated; but if dilute, nearly the whole of the zinc separates as a white precipitate. Chloride of ammonium does not precipitate the solution of hydrate of zinc in potash.

3. *Ammonia** produces in neutral solutions the same precipitate, which readily dissolves in excess of the precipitant. The concentrated solution becomes turbid when mixed with water. On boiling the concentrated solution part of the zinc separates; on boiling the dilute solution all the zinc separates.

4. *Carbonate of sodium** produces a precipitate of basic carbonate of zinc ($3ZnH_2O_2 . 2ZnCO_3$), which is insoluble in excess of the precipitant. Presence of ammonium salts in great excess prevents the formation of this precipitate.

5. *Carbonate of ammonium** produces the same precipitate. It dissolves in excess of the precipitant.

6. *Ferrocyanide of potassium* throws down ferrocyanide of zinc (Zn_2Cy_6Fe) as a white slimy precipitate, somewhat soluble in excess of the precipitant, insoluble in hydrochloric acid.

7. *Ferricyanide of potassium* throws down ferricyanide of

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* If the solution contains non-volatile organic acids, the precipitation by alkalies and alkaline carbonates is hindered. Presence of sugar does not interfere with the precipitation.

zinc $[Zn_3(Cy_6Fe)_2]$ as a brownish orange-colored precipitate, soluble in hydrochloric acid and ammonia.

8. *Carbonate of barium* precipitates no zinc in the cold, if sulphuric acid is absent.

Chromium.

DRY REACTIONS.

1. Compounds of chromium color the *borax bead.* After being heated in the outer or inner blowpipe flame, the bead is emerald color when cold.

2. Compounds of chromium fused with *chlorate of potassium and carbonate of sodium* on a fragment of porcelain, yield a yellow mass from the formation of chromate of sodium (Na_2CrO_4).

WET REACTIONS.

The solutions have sometimes a green, sometimes a violet color.

3. *Sulphide of ammonium* produces a precipitate of hydrate of chromium (CrH_3O_3), thus:—

$$2CrCl_3+3(NH_4)_2S+6H_2O=2CrH_3O_3+6NH_4Cl+3H_2S.$$

4. *Potash* produces in green or violet solutions a bluish-green precipitate of hydrate of chromium, which dissolves readily in an excess of the precipitant to an emerald-green fluid. Upon long boiling, the whole of the hydrate of chromium separates again from this solution, and the supernatant fluid becomes colorless. The same reprecipitation takes place if chloride of ammonium is added to the solution in potash; application of heat promotes the separation of the precipitate.

5. *Ammonia* produces in green solutions a greyish-green, in violet solutions a greyish-blue precipitate of hydrate of chromium. The former precipitate dissolves in acids to a green fluid, the latter to a violet fluid. Other circumstances (concentration, way of adding the ammonia, &c.) exercise also some influence upon the composition and color of the precipitate. A small portion of the hydrate dissolves in excess of the precipitant in the cold, imparting a pink tint to the fluid, but if after the addition of ammonia in excess the mixture is heated, the precipitation will be complete.

6. *Carbonate of ammonium* precipitates basic carbonate of chromium, which redissolves in a large excess of the precipitant.

7. *Carbonate of barium* precipitates all the chromium as hydrate mixed with basic salt. The precipitation takes place in the cold, but is complete only after long digestion.

8. If *chloride of soda* is added to a solution of chromium and the mixture is boiled, the liquid turns yellow from the formation of chromic acid, thus:—

$$2CrCl_3 + 3Na_2Cl_2O + 5H_2O = 2H_2CrO_4 + 6NaCl + 6HCl.$$

Aluminium.

WET REACTIONS.

1. *Sulphide of ammonium* throws down a bulky precipitate of hydrate of aluminium (AlH_3O_3).

2. *Potash* throws down hydrate of aluminium, which contains potash and generally also some basic salt. The precipitate dissolves readily in excess of the precipitant. From this solution in potash the aluminium is precipitated by addition of chloride of ammonium and warming. Presence of ammonium salts does not prevent the precipitation by potash.

3. *Ammonia* produces a precipitate of hydrate of aluminium, which contains ammonia and some basic salt. The precipitate redissolves in a very considerable excess of the precipitant, but with difficulty only. Boiling favors the precipitation.

4. *Carbonate of ammonium* precipitates basic carbonate of aluminium, which is slightly soluble in excess of the precipitant. Boiling favors the precipitation.

5. *Carbonate of barium* precipitates the aluminium upon digestion in the cold as hydrate mixed with basic salt.

6. *Phosphate of sodium* precipitates phosphate of aluminium ($AlPO_4$). The precipitate is bulky and dissolves readily in potash, but not in ammonia; chloride of ammonium therefore precipitates it from its solution in potash. The precipitate dissolves readily in hydrochloric or nitric acid, but not in acetic acid (here it differs from the hydrate), therefore acetate of sodium precipitates it from its solution in hydrochloric acid, when there is not too much of the latter.

GROUP IV.

BARIUM. STRONTIUM. CALCIUM.

Barium.

DRY REACTION.

1. Barium compounds color *flame* yellowish-green.

WET REACTIONS.

2. *Carbonate of ammonium* throws down carbonate of barium ($BaCO_3$) in the form of a white precipitate. If the solution was previously acid, complete precipitation only takes place upon heating the fluid. In chloride of ammonium the precipitate is soluble to a trifling yet clearly perceptible extent; carbonate of ammonium therefore produces no precipitate in very dilute solutions of barium containing much chloride of ammonium.

3. *Oxalate of ammonium* produces a white pulverulent precipitate of oxalate of barium ($BaC_2O_4 + aq.$), which is soluble in hydrochloric and nitric acids. When recently thrown down, this precipitate dissolves in oxalic and acetic acids, but the solutions speedily deposit acid oxalate of barium [$BaH_2(C_2O_4)_2 + 2aq.$] in the form of a crystalline powder.

4. *Phosphate of sodium* produces a white precipitate of phosphate of barium ($BaHPO_4$), which is soluble in acids. Chloride of ammonium dissolves the precipitate to a clearly perceptible extent.

5. *Sulphate of calcium* produces, even in very dilute solutions, a heavy, finely divided white precipitate of sulphate of barium ($BaSO_4$), which is insoluble in alkalies, nearly so in dilute acids, but perceptibly soluble in strong boiling hydrochloric and nitric acids, and in concentrated solutions of ammonium salts. It is, however, insoluble in ammonium salts in presence of excess of sulphuric acid or sulphates. From highly dilute solutions, especially if strongly acid, the precipitate may take some time to form.

6. *Sulphuric acid* produces the same precipitate.

7. *Bichromate of potassium* gives a light yellow precipitate of chromate of barium ($BaCrO_4$), even in very dilute solutions.

It dissolves readily in nitric and hydrochloric acids, and is reprecipitated by ammonia. The neutral chromate of potassium behaves like the bichromate.

8. *Hydrofluosilicic acid* throws down silicofluoride of barium ($BaSiF_6$) in the form of a colorless crystalline quickly-subsiding precipitate. In dilute solutions the precipitate is formed only after the lapse of some time; it is perceptibly soluble in hydrochloric and nitric acids. Addition of an equal volume of alcohol hastens the precipitation and makes it so complete that the filtrate remains clear upon addition of sulphuric acid.

Strontium.

DRY REACTION.

1. Compounds of strontium color *flame* intensely red.

WET REACTIONS.

2. *Carbonate of ammonium* precipitates carbonate of strontium ($SrCO_3$). The precipitate is even less soluble than carbonate of barium in chloride of ammonium.

3. *Oxalate of ammonium* produces a white precipitate of oxalate of strontium ($2SrC_2O_4 + 5aq.$), which dissolves readily in hydrochloric and nitric acids. It is perceptibly soluble in salts of ammonium, and sparingly soluble in oxalic and acetic acids.

4. *Phosphate of sodium* precipitates phosphate of strontium.

5. *Sulphate of calcium* produces a precipitate of sulphate of strontium ($SrSO_4$). Even in strong solutions of strontium the precipitate takes some time to form. The precipitate dissolves perceptibly in hydrochloric and nitric acids. Presence of considerable quantities of these acids most seriously impair the delicacy of the reaction.

6. *Sulphuric acid* produces the same precipitate. When precipitated from concentrated solutions by dilute sulphuric acid, it is at first amorphous and flocculent, then pulverulent and crystalline; when precipitated from dilute solutions by dilute sulphuric acid or by sulphates, it is at once pulverulent and crystalline. Heat favors the precipitation considerably. Addition of alcohol also promotes its separation. Sulphate of

strontium is insoluble in a concentrated solution of sulphate of ammonium even upon boiling.

7. *Bichromate of potassium* does not precipitate solutions of strontium even when concentrated. The neutral chromate at first occasions no precipitation; but on long standing, if the solution is not very dilute, a light yellow crystalline precipitate of chromate of strontium separates. The precipitate is slightly soluble in water, and readily soluble in hydrochloric, nitric, and chromic acids.

8. *Hydrofluosilicic acid* fails to produce a precipitate even in strong solutions of strontium. Upon addition of an equal volume of alcohol no precipitation takes place, except in very concentrated solutions.

Calcium.

DRY REACTION.

1. Compounds of calcium color *flame* yellowish-red.

WET REACTIONS.

2. *Carbonate of ammonium* precipitates carbonate of calcium ($CaCO_3$). When recently precipitated it is bulky and amorphous; after a time, and immediately upon application of heat, it shrinks and becomes crystalline. When recently precipitated it dissolves pretty readily in chloride of ammonium; but the solution soon becomes turbid, and deposits the greater part of the dissolved salt in the crystalline form.

3. *Oxalate of ammonium* precipitates oxalate of calcium as a white powder. If the solution is concentrated or hot the precipitate consists of $CaC_2O_4 + aq.$, and falls at once; but if the solution is very dilute and cold, the precipitate consists of $CaC_2O_4 + aq.$ mixed with $CaC_2O_4 + 3aq.$, and forms only after some time. Oxalate of calcium dissolves readily in hydrochloric and nitric acids, but acetic and oxalic acids fail to dissolve it to any perceptible extent.

4. *Phosphate of sodium* precipitates phosphate of calcium.

5. *Sulphate of calcium* produces no precipitate in solutions of calcium.

6. *Sulphuric acid* produces immediately in strong solu-

tions of calcium a white precipitate of sulphate of calcium ($CaSO_4 . H_2O + aq.$), which redissolves in much water and is far more soluble in acids. No precipitation takes place in dilute solutions until addition of alcohol, when a precipitate forms at once. Sulphate of calcium dissolves readily in a strong solution of sulphate of ammonium on boiling.

7. *Bichromate and neutral chromate of potassium* are without action on salts of calcium.

8. *Hydrofluosilicic acid* does not precipitate salts of calcium.

GROUP V.

MAGNESIUM. POTASSIUM. SODIUM. AMMONIUM.

Magnesium.

WET REACTIONS.

1. *Ammonia* throws down from neutral solutions part of the magnesium in the form of hydrate (MgH_2O_2); the precipitate is soluble in chloride of ammonium.

2. *Potash* throws down hydrate of magnesium; the separation is promoted by boiling.

3. *Baryta* produces the same precipitate.

4. *Lime* produces the same precipitate.

5. *Carbonate of ammonium* produces no precipitate in presence of chloride of ammonium, except in very strong solutions.

6. *Oxalate of ammonium* produces no precipitate in dilute solutions. In rather strong solutions crystalline crusts of various oxalates of magnesium and ammonium make their appearance after some time. In very strong solutions oxalate of ammonium speedily produces a precipitate of oxalate of magnesium ($MgC_2O_4 + 2aq.$) mixed with small quantities of oxalates of magnesium and ammonium. Chloride of ammonium, especially in presence of free ammonia, interferes with the formation of these precipitates, but will not in general prevent it.

7. *Phosphate of sodium* precipitates phosphate of magnesium ($MgHPO_4 + 7aq.$).

8. If chloride of ammonium and ammonia are added to solution of magnesium no precipitation takes place. If, then, phosphate of sodium is added, a crystalline precipitate of

phosphate of magnesium and ammonium ($MgNH_4PO_4+6aq.$) falls, thus:—

$$MgSO_4+NH_4HO+Na_2HPO_4=MgNH_4PO_4+Na_2SO_4+H_2O.$$

The separation of the precipitate is promoted by stirring with a glass rod. If the solution is too dilute for any precipitate to form in the usual manner, yet the lines in which the end of the rod has moved on the vessel will after some time appear distinctly as white streaks. The streaks may be known from scratches by their solubility in hydrochloric acid. Water and ammonium salts dissolve the precipitate but very slightly. It is readily soluble in acids, even in acetic acid. In water containing ammonia it may be said to be insoluble. This reaction is exceedingly delicate.

Potassium.

DRY REACTION.

1. Compounds of potassium color *flame* violet.

WET REACTIONS.

2. *Chloride of platinum* produces in acid and neutral solutions a heavy yellow precipitate of chloride of potassium and platinum (K_2PtCl_6), consisting of microscopic octahedrons. The experiment is conveniently performed in a watch-glass. In strong solutions the precipitate forms immediately; in dilute solutions it may take a considerable time to form; very dilute solutions are not precipitated. Alkaline solutions should be acidified with hydrochloric acid previous to the addition of the reagent. The precipitate is difficultly soluble in water; the presence of free acids does not increase its solubility; it is insoluble in alcohol.

The most delicate way of testing for potassium is to evaporate the solution to be tested with the reagent nearly to dryness on a water-bath, and to treat the residue with a little water (or spirit), when the precipitate will remain undissolved.

3. *Tartaric acid* produces in strong neutral solutions a white crystalline precipitate of acid tartrate of potassium ($KHC_4H_4O_6$), which quickly settles. Shaking and stirring promote the precipitation. Dilute solutions are not precipitated.

Sodium.

DRY REACTION.

1. Sodium compounds color *flame* intensely yellow.

WET REACTIONS.

2. *Chloride of platinum* produces no precipitate in acid or neutral solutions.

3. *Tartaric acid* produces no precipitate.

Ammonium.

DRY REACTION.

1. Ammonium compounds are *volatile.*

WET REACTIONS.

2. *Chloride of platinum* produces in acid and neutral solutions a heavy yellow precipitate of chloride of ammonium and platinum [$(NH_4)_2PtCl_6$], consisting of microscopic octahedrons. Weak solutions are not precipitated.

3. *Tartaric acid* produces in strong neutral solutions a white crystalline precipitate of acid tartrate of ammonium ($NH_4HC_4H_4O_6$).

4. If an ammonium salt is heated with *potash* or *lime,* ammonia is given off, which may be recognised by its smell or by its action on test-paper.

For general purposes it is best to put a small quantity of the substance to be tested (whether solid or liquid it does not matter) in a test tube, to add a c.c. of potash, and warm with the thumb on the mouth of the tube, then to sniff the gaseous contents of the tube up one nostril, the other being closed.

For detecting minute quantities of ammonium, put the substance in a small beaker with a little dry hydrate of calcium and a little water, cover the beaker with a watch-glass having a slip of moist turmeric paper adhering to it, and apply a gentle heat. The escaping ammonia will color the paper brown. With very small quantities of ammonia this reaction takes some time.

For detecting traces of ammonia, the following two reactions may be recommended.

5. When to a neutral solution containing a trace of an ammonium salt a drop of *mercuric chloride* is added, and then a few drops of *carbonate of sodium*, a precipitate consisting of $NH_2HgCl . HgO$ falls, thus—

$$2NH_4HO + 4HgCl_2 + 3Na_2CO_3 = 2(NH_2HgCl . HgO) \\ + 6NaCl + 3H_2O + 3CO_2$$

Of course the quantities of the mercury and sodium salts must not be enough to produce a yellow precipitate of themselves.

6. *Nessler's Test.* If Nessler's solution (p. 11) is added to a fluid containing an ammonium salt, a brown precipitate or yellow coloration is produced according to the quantity of ammonia present. The precipitate consists of $NHg_2I . H_2O$. The change may be expressed thus:—

$$2K_2HgI_4 + 3KHO + NH_4HO = NHg_2I . H_2O + 7KI + 3H_2O$$

SEPARATION OF THE METALS.

Referring to page 25, we will suppose that a solution containing all the common metals has been treated as there indicated, and that thus the several metallic constituents have been separated into groups.

We shall now show how in each of these groups the individual members may be separated from each other, and detected.

Group I.

Precipitated by hydrochloric acid.

The precipitate is washed: it contains—

Silver chloride ($AgCl$).
Mercurous chloride ($HgCl$).
Lead chloride ($PbCl_2$).

It is treated three times on the filter with hot water.

The filtrate contains the lead chloride. It is mixed with sulphuric acid; a white precipitate of sulphate of lead indicates *lead.*

The residue is washed with boiling water. It contains the mercurous and silver chlorides. It is treated with ammonia and filtered.

The filtrate* contains the silver. It is mixed with excess of nitric acid when the chloride of *silver* will be reprecipitated.

The residue is black. This indicates the presence of *monad mercury.*

The lead is not completely precipitated in this group. Small quantities, therefore, always remain in solution; they are completely thrown down by the sulphuretted hydrogen in the next group.

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* This is sometimes turbid from presence of a basic salt of lead. The turbidity may be disregarded as it is soluble in nitric acid.

Group II.

Precipitated by hydrosulphuric acid, in the acid solution, after removal of Group I.*

The precipitate is washed : it contains—

Division I.

Lead sulphide (PbS).
Mercuric sulphide (HgS).
Bismuth sulphide (Bi_2S_3).
Copper sulphide (CuS).
Cadmium sulphide (CdS).

Division II.

Tin sulphide (SnS or SnS_2).
Antimony sulphide (Sb_2S_3).
Arsenic sulphide (As_2S_3).

It is warmed with yellow sulphide of ammonium, and filtered.

Division I.

The residue is washed. It contains the sulphides of Division I. It is removed to a test-tube, boiled with strong nitric acid, diluted and filtered.

The residue is black. It is the mercuric sulphide. (It may contain also some sulphate of lead.) It is dissolved in strong hydrochloric acid with the addition of a little chlorate of potassium. The solution contains mercuric chloride; on mixing it with stannous chloride, the *dyad mercury* will be precipitated as mercurous chloride or in the metallic state.

The filtrate from the mercuric sulphide contains the lead, bismuth, copper, and cadmium, as nitrates. It is mixed with sulphuric acid, evaporated nearly to dryness on a water-bath, diluted slightly, and filtered.

The residue is sulphate of lead. To prove this, treat it with bichromate of potassium, and warm, which will convert it into chromate of lead; wash this, add a little potash, and heat,

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* For separating we generally pass the gas through the fluid, as adding the solution of hydrosulphuric acid would dilute it too much.

the precipitate will dissolve; on acidifying the solution with acetic acid, the chromate of *lead* will be reprecipitated.

The filtrate from the sulphate of lead is treated with ammonia in excess, and filtered.

The precipitate is hydrate of *bismuth;* this may be proved by dissolving it in a drop or two of hydrochloric acid on a watch-glass, and adding water, which will produce a milkiness.

The ammoniacal filtrate from the bismuth contains only copper and cadmium.

Copper will be indicated generally by the blue color of the fluid. If the solution is very dilute, it may be necessary to concentrate it by evaporation to bring out the color; of course, if all the excess of ammonia has been driven off in the evaporation, more must be added.

The cadmium must be separated from the copper before it can be detected. To this end, the filtrate from the bismuth is mixed with hydrosulphuric acid, and filtered. The precipitate contains both the metals as sulphides; it is boiled with dilute sulphuric acid, and filtered. The residue is sulphide of copper. The filtrate contains the cadmium as sulphate; on adding hydrosulphuric acid to it, the yellow sulphide of *cadmium* will be thrown down.

Division II.

The sulphide of ammonium solution contains the tin, antimony, arsenic, and a little copper. It is mixed with excess of hydrochloric acid, and filtered. The precipitate is washed: it contains all the metals as sulphides. It is dried on the filter, and divided into three portions.

The first portion is tested for tin and antimony as follows. It is boiled with strong hydrochloric acid, diluted a little and filtered. The filtrate contains the tin and antimony; it is transferred to a platinum crucible lid, and a little piece of zinc plate is added to it. The *antimony* will show itself at once by staining the platinum black; the tin will be reduced to the metallic state. On taking out the zinc, washing off any adhering tin back into the platinum crucible lid, and pouring away the solution, the residual *tin* may be dissolved by boiling with strong hydrochloric acid, and may be detected in the solution by the addition of mercuric chloride.

The second portion is examined for *arsenic* by fusing with cyanide of potassium and carbonate of sodium in an ignition-tube.

The third portion may be examined for copper by boiling with a few drops of strong nitric acid, adding ammonia, and filtering if necessary. A blue color will indicate copper.

Group III.

Precipitated by ammonia and sulphide of ammonium in presence of chloride of ammonium after the removal of the previous Groups.

The precipitate is washed: it contains—

Nickel sulphide (NiS).
Cobalt sulphide (CoS).
Iron sulphide (FeS).
Manganese sulphide (MnS).
Zinc sulphide (ZnS).
Chromium hydrate (CrH_3O_3).
Aluminium hydrate (AlH_3O_3).

It is treated with dilute hydrochloric acid, well stirred or shaken (but not heated), and filtered.

The residue is washed. It contains nearly the whole of the nickel and cobalt sulphides. The *cobalt* may be found at once, even if there is a very large proportion of nickel present, by testing a small portion in the borax bead. The nickel must be separated from the cobalt before it can be detected. This is done as follows. The filter containing the residue is dried and incinerated (see p. 21), and the ash is dissolved in a few drops of aqua regia, diluted and filtered. The solution contains the metals as chlorides; it is nearly neutralized with potash, and nitrite of potassium and acetic acid are added, the latter to strongly acid reaction. After standing a long time, the cobalt separates completely in the form of the yellow precipitate. On filtering this off, and adding potash to the filtrate, hydrate of *nickel* is precipitated, which may be tested by the borax bead.

The filtrate from the nickel and cobalt sulphides contains the rest of the metals as chlorides (the iron being in the form of ferrous chloride), and also traces of nickel and cobalt. It is mixed with a little strong nitric acid, and boiled, to convert the ferrous chloride into ferric chloride. It is then nearly neutralized with potash, mixed with excess of carbonate of barium in the cold, and filtered.

The precipitate is well washed. It contains the iron, chromium, and aluminium, as hydrates mixed with basic chlorides, together with the excess of carbonate of barium. It is tested separately for each of the metals. One portion is dissolved in hydrochloric acid, and mixed with sulphocyanide of potassium; a red color indicates *iron*. Another portion is boiled with potash, and filtered; the filtrate contains the *aluminium*, which may be thrown down as hydrate by the addition of excess of chloride of ammonium, and boiling. A third portion is boiled with chloride of soda, and filtered; the filtrate contains the *chromium* as chromic acid, which betrays itself by the yellow* color of the fluid.

The filtrate from the carbonate of barium precipitate contains the zinc and manganese as chlorides, and also chloride of barium. It is mixed with sulphuric acid to precipitate the barium, filtered through charcoal to remove the finely divided sulphate of barium, evaporated to a small bulk, mixed with potash, and filtered.

The precipitate contains the manganese as hydrate, and the traces of nickel and cobalt originally dissolved by the hydrochloric acid. It is tested with a bead of carbonate of sodium and a little nitre before the blowpipe. A green bead indicates *manganese*.

The filtrate from the manganese contains the zinc. It is mixed with hydrosulphuric acid; a white precipitate indicates *zinc*.

Group IV.

Precipitated by carbonate of ammonium in presence of chloride of ammonium, after the removal of the previous Groups.

The precipitate is washed: it contains—

Barium carbonate ($BaCO_3$)

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* If the precipitate produced by carbonate of barium had not been well washed, the filtrate here might have been pink from the formation of permanganic acid. The latter may be destroyed by boiling with alcohol; on filtering off the brown oxide of manganese formed, the yellow tint of chromic acid will be visible.

Strontium carbonate ($SrCO_3$)
Calcium carbonate ($CaCO_3$)

It is dissolved in hydrochloric acid. The solution contains the metals as chlorides. It is evaporated to dryness, treated with alcohol (a pestle being used to bring the fluid well in contact with the chlorides), and filtered.

The residue contains the greater part of the chloride of barium. It is dissolved in water, and mixed with a considerable bulk of sulphate of calcium; an immediate precipitate indicates *barium.*

The alcoholic filtrate from the chloride of barium contains the strontium, calcium, and traces of barium, as chlorides. It is mixed with sulphuric acid and filtered. The precipitate contains all the metals as sulphates. It is boiled with a concentrated solution of sulphate of ammonium and a little ammonia for some time, and then filtered.

The residue consists of sulphate of *strontium* and the traces of sulphate of barium.

The filtrate contains the sulphate of calcium. It is largely diluted and mixed with oxalate of ammonium; a precipitate indicates *calcium.*

Traces of barium and still smaller traces of calcium may escape precipitation in this Group. The filtrate from the carbonate of ammonium precipitate should therefore be tested for these metals as follows. One portion should be mixed with sulphate of ammonium; a turbidity indicates barium. Another portion should be mixed with oxalate of ammonium, and allowed to stand for some time; a turbidity indicates calcium.

Group V.

Left in solution after the removal of the previous Groups.

As just mentioned, the present solution may contain traces of barium and calcium which should be removed before the examination for the members of this group is proceeded with. This removal is effected by mixing the solution with sulphate and oxalate of ammonium, allowing to stand for some time and filtering.

The solution contains—

Magnesium
Potassium
Sodium
Ammonium.

Besides ammonium salts, added to precipitate the previous groups.

A small portion is mixed with phosphate of sodium, and well stirred; a crystalline precipitate indicates *magnesium.*

The rest of the solution is to be examined for potassium and sodium. It must, however, first be freed from magnesium and ammonium, as follows. It is evaporated to dryness, and ignited till white fumes cease to be evolved (*i.e.,* till all the ammonium salts are driven off). The residue is treated with a little water, warmed, and mixed with milk of lime* (or baryta water) till the reaction is strongly alkaline, boiled and filtered. The magnesium is thus removed. The filtrate containing the potassium and sodium and excess of lime (or baryta) is mixed with carbonate of ammonium, gently warmed, and filtered. The filtrate is evaporated with a little chloride of ammonium (to convert alkalies or alkaline carbonates into chlorides) to dryness, gently ignited, and dissolved in a *little* water.

The solution contains the potassium and sodium in a concentrated form. One portion is mixed with chloride of platinum; a precipitate indicates *potassium.* Another portion is tested in the Bunsen flame on a clean platinum wire; a yellow flame indicates *sodium.*

It is useless to test for ammonium with the other members of this group, as the solution has been mixed with ammonium salts in the precipitation of the previous groups. For this purpose, then, some of the original solution is boiled with potash; a smell of ammonia indicates *ammonium.*

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* Freed from alkalies by repeated boiling with fresh quantities of water.

EXTRA METHODS OF SEPARATING METALS.

Group II.

Division II.

Separation of Antimony, Arsenic, and Tin.

1. The mixed *sulphides* are dried, mixed with one part of pure dry carbonate of sodium and three parts of nitrate of sodium, and fused in a porcelain crucible at a gentle heat. The heat must not be too strong, nor must the fusion be continued too long, or some hydrate of sodium may be produced, and a soluble stannate of sodium formed. The fused mass is poured out onto a clean stone and powdered finely. It contains the tin as stannic oxide, the antimony as antimoniate of sodium, the arsenic as arseniate of sodium, the excess of carbonate and nitrate of sodium, and also sulphate and nitrite of sodium. It is treated with cold water and filtered.

The filtrate contains the arseniate of sodium, and the carbonate, nitrate, sulphate, and nitrite of sodium. In order to detect the *arsenic*, it is acidified with nitric acid, warmed to remove carbonic anhydride, mixed with nitrate of silver, and filtered, if necessary, from chloride of silver. On inclining the test-tube and pouring some ammonia diluted with ten parts of water down the side onto the top of the solution, a red cloud of arseniate of silver will be seen where the two fluids meet.

The residue is washed with dilute spirit (1 alcohol, 3 water). It contains the stannic oxide and the antimoniate of sodium. It is transferred to a platinum crucible lid; on adding a piece of zinc and some hydrochloric acid, and gently warming for a little time, the tin and antimony will be more or less reduced to the metallic state.

The *antimony* will at once betray its presence by blackening the platinum.

The *tin* is detected by removing the excess of zinc, pouring off the fluid, boiling the residue with strong hydrochloric acid, diluting and filtering. The filtrate contains stannous chloride; on mixing it with mercuric chloride in excess, a white precipitate of mercurous chloride is formed, indicating the presence of *tin*.

2. The mixed *sulphides* are freed from the greater quantity of adhering water by laying the filter containing them on blotting paper, then transferred to a test-tube and treated with strong hydrochloric acid at a gentle heat. The sulphides of antimony and tin dissolve, while the sulphide of arsenic is left almost completely undissolved.

If the residue is considerable, it is dissolved in a few drops of strong nitric acid, and mixed with nitrate of silver, and then some very dilute ammonia is poured onto the top of the solution. If it is very minute, it may be dissolved in ammonia, evaporated to dryness with carbonate of sodium, mixed with cyanide of potassium, and heated in a current of carbonic acid (p. 41, 2).

The solution containing the antimony and tin is put into a platinum crucible lid with zinc, the metals being detected as described in 1. Or if a large excess of antimony is present, the solution may be mixed with an excess of solution of common solid carbonate of ammonium and boiled. The tin will be precipitated with a little of the antimony, and may be more readily detected by the method described in 1.

3. The mixed *sulphides* are digested at a gentle heat with some common solid carbonate of ammonium and water; the sulphide of arsenic dissolves, while the sulphides of antimony and tin remain undissolved. This separation, however, is not absolute, as traces of antimony are apt to pass into solution while a little sulphide of arsenic remains in the residue.

The solution is acidified with hydrochloric acid, which reprecipitates the sulphide of arsenic. If the latter consists only of a few flakes, it should be washed, dissolved in ammonia, evaporated with carbonate of sodium, and heated with cyanide of potassium in a stream of carbonic acid (p. 41, 2.)

The residue insoluble in carbonate of ammonium is dissolved in hydrochloric acid and treated as in 1.

4. The *sulphides* are dissolved in sulphide of potassium, then treated with a large excess of strong solution of sulphurous acid, digested for some time on the water-bath, boiled till all sulphurous acid is expelled, and filtered.

The filtrate contains all the arsenic, as arsenious acid, which may be precipitated by hydrosulphuric acid.

The residue containing the antimony and tin sulphides is dissolved in strong hydrochloric acid and treated as in 1.

5. The *oxides* are fused with hydrate of sodium in a silver crucible; the mass is treated with water, and then alcohol is added to the amount of one-third the volume of the water. The mixture is filtered.

The residue is washed with weak spirit mixed with a few drops of carbonate of sodium. It contains the antimony in the form of antimoniate of sodium. In the presence of much tin it is advisable to subject the residue to a repetition of the above treatment, in order to separate the whole of the tin.

The filtrate contains the tin and arsenic. It is acidified with hydrochloric acid, and the metals are then precipitated by hydrosulphuric acid with the aid of heat. On heating the precipitate in a stream of sulphuretted hydrogen the whole of the tin is left as sulphide, while the sulphide of arsenic volatilizes, and may be received in ammonia.

Separation of Antimony and Arsenic when they are evolved as hydrogen compounds.

One method has been given, p. 46. Another method is as follows:—

The gas is passed slowly through a tube containing broken glass moistened with dilute solution of acetate of lead, to retain hydrochloric and hydrosulphuric acids, and then into solution of nitrate of silver. The antimony in the gas is almost completely precipitated as black antimonide of silver ($SbAg_3$), while the arsenic passes into solution as arsenious acid, metallic silver being thrown down at the same time. The mixture is filtered.

The solution contains the arsenic as arsenious acid, a little of the antimony, nitric acid, and the excess of nitrate of silver. It is mixed with hydrochloric acid and filtered to remove silver, then treated with hydrosulphuric acid. The precipitate containing the arsenic and traces of antimony is fused with carbonate of sodium and cyanide of potassium as directed, p. 41, 2.

The residue is thoroughly freed from the arsenious acid by being boiled with water. It contains the antimonide of silver mixed with silver. It is boiled with tartaric acid solution, and filtered. The filtrate contains the antimony, which is detected by adding hydrochloric acid and hydrosulphuric acid.

Group III.

Separation of Nickel and Cobalt.

The slightly acid solution is mixed with hydrocyanic acid and potash* till the precipitate which first forms is redissolved. The solution contains now cyanide of nickel and potassium (NiK_2Cy_4), and cyanide of cobalt and potassium (CoK_2Cy_4), with excess of potash and hydrocyanic acid. It is boiled for some minutes; if a precipitate forms, more hydrocyanic acid and potash are added. The solution contains now the cyanide of nickel and potassium unaltered, and cobalticyanide of potassium (K_3CoCy_6). It is mixed with excess of chloride of soda, boiled again (the solution should be alkaline), and filtered. The precipitate is washed; it consists of sesquioxide of nickel, and is to be tested in the borax bead. The filtrate contains the cobalt as cobalticyanide of potassium.

Group IV.

Separation of Calcium and Strontium.

The carbonates are dissolved in nitric acid and evaporated to dryness. The residue is powdered, digested for some time with absolute alcohol and ether, and filtered.

The residue is nitrate of strontium. It is dissolved in a little water, and mixed with sulphate of calcium.

The filtrate contains the calcium as nitrate; it is precipitated with sulphuric acid. The precipitate when treated with water should give a solution, which is precipitated at once by oxalate of ammonium.

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* Hydrocyanic acid and potash are used instead of cyanide of potassium, as the latter is often too impure.

Separation of Barium, Strontium, and Calcium, when mixed as Sulphates.

The sulphates are digested in the cold for twelve hours with carbonate of ammonium, or boiled for ten minutes with a solution of one part of carbonate and three parts of sulphate of potassium. The mixture is then filtered. The residue is washed. It contains the strontium and calcium in the form of carbonates, and the barium as the original sulphate, with the exception of a minute trace. On treating the residue with hydrochloric acid, the strontium, calcium, and trace of barium dissolve, while the sulphate of barium remains behind, and may be fused with carbonate of potassium and sodium.

Decomposition of the Phosphates of Barium, Strontium, and Calcium.

The phosphates are dissolved in hydrochloric acid; the solution is nearly neutralized, mixed with ferric chloride and excess of acetate of sodium, and boiled. The mixture is then filtered hot. The precipitate contains the phosphoric acid. The filtrate contains the barium, strontium, and calcium.

Detection of traces of Barium and Strontium in presence of a large amount of Calcium.

Ignite two or three grammes of the mixed carbonates for a few minutes in a platinum crucible over the bellows blowpipe,* boil the residue with a little water, filter, evaporate the filtrate with hydrochloric acid to dryness, and examine the residue by the spectroscope.

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* The carbonates of barium and strontium are reduced to the caustic state much more readily in the presence of carbonate of calcium than they otherwise would be.

REACTIONS OF THE ACIDS.

ARRANGEMENT OF THE ACIDS.

The acids may be arranged as follows :—

ACIDS WHICH ARE PRECIPITATED BY NITRATE OF SILVER IN PRESENCE OF NITRIC ACID—

- Hydrosulphuric acid.
- Hydrochloric acid.
- Hydrobromic acid.
- Hydriodic acid.

ACIDS WHOSE SALTS DEFLAGRATE* ON CHARCOAL—

- Nitric acid.
- Chloric acid.

ACIDS WHICH CANNOT BE CLASSED—

- Boracic acid.
- Carbonic acid.
- Chromic acid.
- Hydrofluoric acid.
- Phosphoric acid.
- Silicic acid.
- Sulphuric acid.

Hydrosulphuric Acid.

DRY REACTION.

1. Some sulphides when heated in an *ignition-tube* give a sublimate of sulphur.

WET REACTIONS.

2. *Nitrate of silver* gives a precipitate of sulphide of silver (Ag_2S) insoluble in dilute acids.

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* To deflagrate is to burn suddenly and with sparks.

3. *Acetate of lead* gives a precipitate of sulphide of lead (PbS), insoluble in dilute acids.

4. Many sulphides, when heated with dilute *hydrochloric acid*, evolve hydrosulphuric acid, which may be recognised by its characteristic smell, and by its blackening a piece of paper moistened with acetate of lead.

Certain sulphides, as iron pyrites and copper pyrites, which are not readily acted on by dilute hydrochloric acid alone, will give abundance of hydrosulphuric acid on the addition of a piece of *zinc*.

Hydrochloric Acid.

WET REACTIONS.

1. *Nitrate of silver* produces a white precipitate of chloride of silver ($AgCl$), which upon exposure to light turns first violet and then black. It is insoluble in dilute nitric acid, but dissolves readily in ammonia and in cyanide of potassium. It fuses without decomposition when heated.

2. *Acetate of lead* produces a precipitate of chloride of lead ($PbCl_2$). It is soluble in a large amount of water, especially if boiling.

3. *Strong sulphuric acid*, heated with dry chlorides, causes an evolution of hydrochloric acid gas which has a pungent smell and gives white fumes in the air. Chlorides of mercury and tin are not thus decomposed.

4. *Binoxide of manganese and sulphuric acid*, heated with a chloride, cause an evolution of chlorine, which may be readily recognised by its odor, its color, and its bleaching action upon moist litmus paper.

5. *Bichromate of potassium and strong sulphuric acid*, heated gently in a retort with a dry chloride, cause an evolution of chlorochromic acid (CrO_2Cl_2), which appears as a brownish-red gas, and may be condensed in a receiver. On mixing chlorochromic acid with ammonia in excess, a yellow-colored liquid is obtained, thus—

$$CrO_2Cl_2 + 4NH_4HO = (NH_4)_2CrO_4 + 2NH_4Cl + 2H_2O.$$

6. Insoluble chlorides are fused with carbonate of potassium and sodium. On treating the fused mass with water, the chlorine dissolves in the form of alkaline chloride. Chloride of silver may be conveniently decomposed by allowing it to stand

for some time in contact with zinc and dilute sulphuric acid; the silver will be reduced, and the solution will contain chloride and sulphate of zinc.

Hydrobromic Acid.

WET REACTIONS.

1. *Nitrate of silver* produces a yellowish-white precipitate of bromide of silver ($AgBr$), which turns grey upon exposure to light. It is insoluble in dilute nitric acid, and somewhat sparingly soluble in ammonia, but dissolves with facility in cyanide of potassium.

2. *Nitrate of palladium* (but not chloride of palladium) produces in neutral solutions a reddish-brown precipitate of bromide of palladium ($PdBr_2$). In strong solutions it is formed immediately; in dilute solutions, it does not appear till after some time.

3. If a solution of a bromide is mixed with hydrochloric acid, and a drop or two of *chloride of soda** is then added, bromine will be liberated and tinge the fluid yellow. On adding enough bisulphide of carbon to form a large drop at the bottom of the tube, closing the mouth of the latter with your thumb and shaking well, the bromine will be dissolved by the bisulphide and the color will be more apparent. With 1 part of bromine in 1000 parts of water, the drop of bisulphide acquires a reddish-yellow tint; with 1 part of bromine in 30,000 parts of water, a pale yellow tint will be distinctly discernible.

Excess of chloride of soda must be avoided, as the chlorine formed would recombine with the bromine and prevent it from coloring the bisulphide.

If the solution of bromine in bisulphide of carbon is mixed with potash shaken and warmed, the color disappears, and the aqueous solution now contains the bromine in the form of bromide and bromate of potassium. By evaporation and ignition the whole is converted into bromide of potassium, and may be tested by 6.

4. If a solution of a bromide is mixed with hydrochloric acid and *nitrite of potassium* is added, no bromine is liberated.

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* Chloride of soda and hydrochloric acid may be considered equivalent to chlorine water.

5. *Strong sulphuric acid* heated with a dry bromide causes an evolution of bromine.

6. *Binoxide of manganese and sulphuric acid,* heated with a bromide, cause the evolution of bromine. The presence of chlorides interferes with this reaction; their influence may be lessened by adding water and reducing the quantity of sulphuric acid. Traces of bromine are best detected in this way by gently heating the mixture in a small beaker, covered with a watch glass, to which a slip of paper moistened with starch paste is attached. The starch will be colored yellow.

7. *Bichromate of potassium and sulphuric acid* distilled with a bromide yield bromine, which appears as a brownish-red gas and condenses in the receiver. It may be known from chlorochromic acid by giving a colorless fluid when supersaturated with ammonia.

8. Insoluble bromides are treated like insoluble chlorides. Bromide of silver may be decomposed like chloride of silver.

Hydriodic Acid.

WET REACTIONS.

1. *Nitrate of silver* produces a yellowish-white precipitate of iodide of silver (AgI), which blackens on exposure to light. It is insoluble in dilute nitric acid, very sparingly soluble in ammonia, readily soluble in cyanide of potassium.

2. *Chloride of palladium* (and nitrate of palladium) produces a dark brown precipitate of iodide of palladium (PdI_2), which dissolves to a trifling extent in saline solutions, but is insoluble in dilute hydrochloric and nitric acids.

3. If a solution of an iodide is mixed with hydrochloric acid, and a drop or two of *chloride of soda* is then added, iodine will be liberated. On adding enough bisulphide of carbon to form a large drop at the bottom of the tube, closing the mouth of the latter with your thumb and shaking well, the liberated iodine will be taken up by the bisulphide, which will acquire a beautiful purple color.

If instead of the bisulphide some starch solution* is added, the fluid acquires a fine blue color. This color is destroyed

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* Made by shaking a little starch with plenty of water, and boiling.

by heat, therefore the fluid should be cold. The delicacy of this reaction with starch is impaired by the presence of certain salts, such as alkaline sulphates, &c.

Excess of chloride of soda must be avoided, as the chlorine would recombine with the iodine, and prevent it from giving a color with either the bisulphide or the starch.

4. If a solution of an iodide is mixed with hydrochloric acid, and a drop or two of *nitrite of potassium* is added, the iodine is liberated, and may be detected by bisulphide of carbon or solution of starch, as in 3.

Excess of nitrite of potassium does no harm. This test is therefore preferable to 3.

5. *Strong sulphuric acid* heated with dry iodides causes an evolution of purple vapors of iodine.

6. *Binoxide of manganese and sulphuric acid* heated with iodides cause an evolution of iodine.

7. *Bichromate of potassium and sulphuric acid* heated with iodides cause an evolution of iodine.

8. If *sulphate of copper* is mixed with a neutral solution of an iodide, and then sulphurous acid is added till the brown color produced by the liberated iodine is removed, the iodine is thrown down as cuprous iodide (CuI) in the form of a dirty-white precipitate.

$$2CuSO_4 + 2KI + H_2SO_3 + H_2O = 2CuI + K_2SO_4 + 2H_2SO_4.$$

Chlorides and bromides are not precipitated in this manner.

9. Insoluble iodides are treated like insoluble chlorides. The process given for decomposing chloride of silver answers equally well for decomposing iodide of silver.

Nitric Acid.

DRY REACTIONS.

1. If a nitrate is heated on *charcoal*, deflagration takes place; the charcoal burning at the expense of the oxygen in the nitrate.

2. If a mixture of a nitrate with *cyanide of potassium* in powder is heated on platinum-foil, deflagration takes place. Very small quantities of nitrates may be detected in this manner.

WET REACTIONS.

3. *Strong sulphuric acid* heated with nitrates causes an evolution of fumes of nitric acid.

4. If a solution of a nitrate is mixed with a cold strong solution of ferrous sulphate, and the test tube being held in a slanting position, strong sulphuric acid is gently poured down to the bottom, so that it may not mix with the solution, then a purple or brown color will mark the plane of contact of the two fluids. The following expresses the change which takes place:

$$6FeSO_4+3H_2SO_4+2HNO_3=3Fe_2(SO_4)_3+4H_2O+2NO.$$

The nitric oxide dissolving in the excess of ferrous sulphate, gives the color.*

5. *Copper filings and strong sulphuric acid* heated with a nitrate liberate nitric oxide, thus—

$$2KNO_3+Cu_3+4H_2SO_4=K_2SO_4+3CuSO_4+4H_2O+2NO.$$

The nitric oxide mixing with the air in the tube imparts a red color to it, from the formation of nitric peroxide.

6. *Solution of indigo*, boiled with hydrochloric acid, and a solution of a nitrate, is decolorized. When just enough indigo is used to give a blue color before the addition of the nitrate, the reaction is very delicate; it is, however, not characteristic, as several other substances besides nitric acid (chlorine, for instance) decolorize indigo.

7. A little *brucia* dissolved in strong sulphuric acid, when added to a small quantity of a solution of a nitrate, gives a fine red color. This reaction is very delicate.

8. Dissolve one part of *carbolic acid* in four parts of strong sulphuric acid, and add two parts of water. A drop or two of this fluid added to a solid nitrate (*e.g.*, to the residue obtained by evaporating a few drops of well-water containing nitrates) gives a reddish-brown color, from the formation of a nitro-compound. On addition of a drop or two of strong ammonia, this color turns yellow, sometimes passing through a green shade. A very delicate reaction (H. Sprengel).

9. Very minute quantities of nitric acid may be detected

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* A similar reaction is observed in presence of selenious acid, but no mixing the fluids, and allowing to stand, red selenium separates.

by *reducing it first to nitrous acid.* The solution is heated for some time with zinc amalgam, and filtered. On adding sulphuric acid, iodide of potassium, and starch solution, a blue color is produced (see Reactions of nitrous acid).

10. *Free nitric acid may be detected* by evaporating in a porcelain dish to dryness on a water-bath, with quill cuttings. These will be colored yellow.

Chloric Acid.

DRY REACTIONS.

1. Chlorates deflagrate, when heated on *charcoal*, far more violently than nitrates.

2. Heated on platinum-foil with *cyanide of potassium*, chlorates detonate violently. Only minute quantities should be used in this experiment.

WET REACTIONS.

3. A few drops of *strong sulphuric acid* added to a small quantity of a chlorate liberate peroxide of chlorine, thus—

$$3KClO_3 + 2H_2SO_4 = 2ClO_2 + KClO_4 + 2KHSO_4 + H_2O.$$

The peroxide of chlorine colors the sulphuric acid intensely yellow, and betrays itself also by its odor and greenish color. The experiment should be performed in a watch-glass, and application of heat should be avoided, or an explosion might take place.

4. If a solution of a chlorate is colored light-blue with *indigo* solution, and a little dilute sulphuric acid is added, and then *sulphurous acid*, the blue color at once disappears. The cause of this equally delicate and characteristic reaction is that the sulphurous acid reduces the chloric acid, and the product of the reduction decolorizes the indigo.

5. If a chlorate is warmed with *hydrochloric acid*, water, chlorine, and chlorochloric anhydride ($2Cl_2O_5 . Cl_2O_3$), are formed. The test-tube becomes filled with a greenish-yellow gas of a very disagreeable odor, resembling that of chlorine; the hydrochloric acid acquires a greenish-yellow color. If the hydrochloric acid is colored blue with indigo, the presence of a very minute quantity of chlorate will suffice to destroy the color.

6. If a chlorate is mixed with carbonate of sodium and ignited, oxygen is given off, and a chloride remains. On dissolving the residue, acidifying with nitric acid, and adding nitrate of silver, a precipitate of chloride of silver will be obtained.

Boracic Acid.

DRY REACTIONS.

1. Boracic acid tinges the *Bunsen flame* green. Borates, when fused with carbonate of sodium in the flame of the spectroscope, give several characteristic lines.

2. If a substance containing boracic acid is reduced to a fine powder, mixed on a piece of paper with a drop of water and three parts of a *mixture of acid sulphate of potassium and fluoride of calcium,*[*] and exposed in the loop of a platinum wire to the blowpipe flame, fluoride of boron escapes, and the flame is tinged green for a few seconds.

WET REACTIONS.[†]

3. If a solution of an alkaline borate is mixed with hydrochloric acid to slight but distinct acid reaction, and a slip of *turmeric paper* is half dipped into it, and then dried at 100°, the dipped half shows a peculiar red tint. This reaction is very delicate. Care must be taken not to confound the characteristic red coloration with the blackish-brown color which turmeric paper acquires when moistened with strong hydrochloric acid, and dried; nor with the brownish-red color which ferric chloride or a hydrochloric acid solution of molybdate of ammonium gives to turmeric paper, more particularly on drying. By moistening turmeric paper reddened by boracic acid with a solution of an alkali, the color is changed to bluish-black or greenish-black; but a little hydrochloric acid will at once restore the brownish-red color.

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[*] Consisting of 5 parts of acid sulphate of potassium and 1 part of finely powdered fluor spar.

[†] A solution of boracic acid, when boiled, loses acid. If, therefore, a solution is to be concentrated by evaporation, it should be previously mixed with potash.

Carbonic Acid.

WET REACTIONS.

1. *Lime water* produces with carbonic acid a white precipitate of carbonate of calcium ($CaCO_3$). The reagent ought always to be used in excess, as the precipitate is soluble in excess of carbonic acid. The precipitate dissolves in acids with effervescence, and, after the complete expulsion of the carbonic anhydride by boiling, is not reprecipitated by ammonia.

2. *Hydrochloric acid* added to a carbonate produces an evolution of carbonic anhydride accompanied by effervescence. The gas may be recognised by its want of odor, or by holding a rod moistened with lime-water in it, or by pouring it off into a test-tube containing a little lime-water, and shaking.

Chromic Acid.

DRY REACTION.

1. Compounds of chromic acid give an emerald bead with *borax* in both the outer and inner flame. (See p. 56, 1.)

WET REACTIONS.

2. *Hydrosulphuric acid* added to an acidified solution produces ultimately a green coloration, arising from reduction of the chromic acid. At the same time sulphur is separated, which gives a milky appearance, thus—

$$K_2CrO_4 . CrO_3 + 8HCl + 3H_2S = 2KCl + 2CrCl_3 + 7H_2O + S_3.$$

Heat promotes the reaction; in this case, part of the sulphur is converted into sulphuric acid.

3. *Sulphide of ammonium* added in excess to a solution of an alkaline chromate produces a dirty-green precipitate of hydrated chromate of chromium; on boiling, all the chromium separates as green hydrate.

4. *Sulphurous acid* added to an acidified solution reduces the chromic acid, turning the solution green, thus—

$$K_2CrO_4 . CrO_3 + 2HCl + 3H_2SO_3 = Cr_2(SO_4)_3 + 2KCl + 4H_2O.$$

5. *Chloride of barium* produces a light-yellow precipitate of

chromate of barium ($BaCrO_4$), which is soluble in hydrochloric and nitric acids.

6. *Nitrate of silver* produces a dark purple-red precipitate of chromate of silver (Ag_2CrO_4), which is soluble in nitric acid and ammonia. In slightly acid solutions the precipitate consists of bichromate of silver ($Ag_2CrO_4 . CrO_3$).

7. *Acetate of lead* produces a yellow precipitate of chromate of lead ($PbCrO_4$) which is soluble in potash, sparingly soluble in dilute nitric acid, insoluble in acetic acid. An alkaline solution should be acidified with acetic acid.

8. If a very dilute solution of *peroxide of hydrogen*[*] is covered with a layer of ether, and a fluid containing chromic acid is added, the solution acquires a fine blue color. On closing the test-tube with the thumb, and inverting it repeatedly without much shaking, the color is transferred from the solution to the ether. The latter reaction is peculiarly characteristic and delicate; one part of chromate of potassium in 40,000 parts of water will be indicated.[†] The nature of the blue substance is not known. After some time reduction to chromium salt takes place, and the ether loses its color.

9. *Very minute quantities* of chromic acid may be detected by the following methods:—(*a*) To the solution acidified slightly with sulphuric acid add a little tincture of guaiacum (1 part of the resin in 100 of spirit); an intensely blue color is produced, which, however, soon disappears if only traces of chromic acid are present. (*b*) To the solution as neutral as possible add a little dilute decoction of logwood; an intensely black color is produced; when mere traces of chromic acid are present the color is violet-red.

10. If insoluble chromates are fused with carbonate of sodium and nitrate of potassium, alkaline chromate will be formed, which is soluble in water.

For the discovery of traces of chromium present in many minerals, such as serpentine, &c., the reactions in 9 may be used after the mineral has been fused with alkaline carbonate and nitrate.

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[*] This solution may be easily prepared by adding a little powdered peroxide of barium to some dilute hydrochloric acid. In default of peroxide of barium, impure peroxide of sodium may be used, which is obtained by heating a small piece of sodium on a porcelain crucible lid until it takes fire, and letting it burn.

[†] Presence of vanadic acid impairs the delicacy of the reaction.

Hydrofluoric Acid.

WET REACTIONS.

1. *Chloride of calcium* produces a gelatinous precipitate of fluoride of calcium (CaF_2), which is so transparent as at first to induce the belief that the fluid has remained perfectly clear. Addition of ammonia promotes the complete separation of the precipitate. The precipitate is almost absolutely insoluble in water, and only very slightly soluble in hydrochloric and nitric acids in the cold; it dissolves somewhat more largely upon boiling with hydrochloric acid. Ammonia produces no precipitate in the solution, or only a trifling one, as the salt of ammonium formed retains it in solution. Fluoride of calcium is scarcely more soluble in hydrofluoric acid than in water. It is insoluble in alkaline fluids.

2. *Etching test.* If a dry fluoride is warmed in a platinum crucible with a little strong sulphuric acid, and the crucible is covered with a piece of glass, the latter will be found to be corroded from the hydrofluoric acid produced.

The most delicate way of applying this test is as follows:—Take a small piece of glass, warm it, and rub some wax over one side, then allow it to cool, and make a mark on the waxed side with a sharp point. If a finely powdered fluoride is treated in a platinum crucible with just enough strong sulphuric acid to make it into a thin paste, and the crucible is covered with the prepared glass and placed in a warm place for half an hour, the mark will be found, after the removal of the wax, to be etched into the glass. When only a mere trace of fluorine is present, the etching may not appear until the glass is breathed upon.

This reaction fails if there is too much silica present, or if the substance is not decomposed by sulphuric acid. In such cases one or other of the two following methods is resorted to, according to circumstances:—

3. In the case of a *fluoride decomposable by sulphuric acid mixed with a large quantity of silica*, the substance may be heated with strong sulphuric acid in a test-tube, when fluoride of silicon will be evolved, which forms white fumes in the air. If the gas is conducted into water, gelatinous silicic acid will separate in the water.

The following modification answers best for the detection of traces of fluorine. Heat the substance with strong sulphuric acid in a flask closed with a cork bearing two tubes, one of which reaches to the bottom of the flask, whilst the other just passes through the cork. Pass into the flask through the long tube a slow stream of dry air, and conduct the air reissuing from the other tube into a U-tube containing a little ammonia and connected with an aspirator. The fluoride of silicon which escapes with the air decomposes with the ammonia, more particularly if a gentle heat is applied to the U-tube towards the end of the process, fluoride of ammonium and silicic acid being formed. Filter, evaporate in a platinum crucible to dryness, and examine the residue according to 2.

4. *Silicates not decomposable by sulphuric acid* must first be fused with four parts of carbonate of potassium and sodium. The fused mass is treated with water, the solution filtered, the filtrate concentrated by evaporation, allowed to cool, transferred to a platinum vessel, hydrochloric acid added to feebly acid reaction, and the fluid allowed to stand until the carbonic anhydride has escaped. It is then supersaturated with ammonia, heated, filtered into a flask, mixed with chloride of calcium, corked up, and allowed to stand. If a precipitate separates after some time, it is filtered off, dried and examined according to 2.

Phosphoric acid.

WET REACTIONS.

1. If *sulphate of magnesium*, mixed with a sufficient quantity of chloride of ammonium to leave the solution clear upon addition of ammonia, is added to a solution containing phosphoric acid, and then *ammonia* in excess is added, a white crystalline precipitate is formed, which rapidly subsides. The precipitate is phosphate of magnesium and ammonium ($MgNH_4PO_4 + 6aq.$); it is insoluble in ammonia, and most sparingly soluble in chloride of ammonium, but dissolves readily in acids, even in acetic acid. It is sometimes slow in appearing; stirring promotes its separation. This reaction can be considered decisive only if arsenic acid is absent.

2. *Nitrate of silver* throws down from neutral solutions a light-yellow precipitate of phosphate of silver (Ag_3PO_4), readily soluble in nitric acid and ammonia.

3. The *solution of molybdate of ammonium in nitric acid* (see p. 7) gives in the cold a finely-divided yellow precipitate, which speedily subsides. With very minute quantities of phosphoric acid—as, for instance, ·02 mgrm.—a few hours must be allowed for the manifestation of the reaction, and the liquid should be gently warmed, but not above 40°. The fluid above the precipitate appears colorless. Not more than an equal volume of the fluid to be tested should be added to the molybdate solution. A mere yellow coloration of the fluid should never be considered to prove the presence of phosphoric acid.

The yellow precipitate consists of molybdic acid, ammonia, water, and a little phosphoric acid (about three per cent.). As it is insoluble in dilute acids only in presence of an excess of molybdic acid, addition of phosphoric acid in excess may prevent its formation. Presence of large quantities of hydrochloric acid and of certain organic substances (tartaric acid, for instance) interferes with the precipitation. The precipitate after subsiding may be readily recognised in dark-colored fluids. By washing it with the molybdate solution, dissolving in ammonia, and adding chloride of ammonium and sulphate of magnesium, the precipitate of phosphate of magnesium and ammonium may be obtained.

By conducting the operation in the way indicated, phosphoric acid cannot well be confounded with any other acid, since arsenic acid gives no precipitate in the cold with this reagent, though it gives one upon application of heat, and especially upon boiling (the supernatant fluid appearing yellow); and silicic acid shows no reaction in the cold, and gives only a yellow coloration on heating, but no precipitate.

4. *White of Egg* is not precipitated by solution of phosphoric acid, nor by solutions of phosphates mixed with acetic acid.

5. An insoluble substance containing phosphoric acid may be fused in a platinum crucible with three parts of carbonate of sodium, one part of nitrate of potassium, and one part of silica. On boiling the fluid mass with water, decanting the solution, adding carbonate of ammonium to it, boiling, and filtering from the silicic acid, the phosphoric acid will be obtained in the solution in the form of alkaline phosphate.

Silicic Acid.

DRY REACTION.

1. If a fragment of silica or a silicate is heated in a bead of *microcosmic salt*, the silica remains undissolved, and floats about in the bead as a more or less transparent mass (called a skeleton), exhibiting the shape of the original fragment. In the case of a silicate the bases dissolve.

WET REACTIONS.

2. *Chloride of ammonium* produces in not too dilute solutions of alkaline silicates a precipitate of hydrated silica (containing alkali). Heating promotes the separation.

3. The solutions of alkaline silicates are decomposed by all *acids*. If a large proportion of hydrochloric acid is added at once even to concentrated solutions, the separated silica remains in solution; but if hydrochloric acid is added drop by drop while the fluid is stirred, the greater part of the silica separates as gelatinous hydrate.

If the solution, after having been mixed with excess of hydrochloric acid, is evaporated to dryness, and the residue is thoroughly dried by being powdered in the dish with a pestle and heated again, the whole of the silica will be rendered insoluble, and may be separated by treating the residue with hydrochloric acid and water.

DECOMPOSITION OF NATURAL SILICATES.

The silicates must in all cases be very finely powdered, before being subjected to any of these processes.

By treatment with hydrochloric, nitric, or sulphuric acid. Applicable to some silicates. Some silicates are decomposed by hydrochloric or nitric acid. To effect the complete separation of the silica, the mixture of silicate and acid is evaporated to dryness, heated at a temperature rather above the boiling point of water until no more acid fumes escape, lumps being broken up with a pestle so that the whole may be completely dried. The powder is then moistened with hydrochloric acid, heated with water, and filtered. The filtrate contains the metals, and the residue contains all the silica.

Some silicates, kaolin for instance, not decomposable by hydrochloric or nitric acid, are completely decomposed by heating with a mixture of eight parts of strong sulphuric acid and three parts of water, the silica being separated in the form of a powder.

Silicates not decomposable by boiling with hydrochloric or sulphuric acid in the open air, may generally be completely decomposed by heating with the acids in sealed glass tubes at 200°—210° in an air or paraffin bath.

By treatment with hydrofluoric acid. Applicable to all silicates. If a silicate is mixed with three parts of fluoride of ammonium or five parts of fluoride of calcium in powder, and then made into a paste with strong sulphuric acid, and heated in the open air until no more acid fumes escape, the whole of the silica passes off as fluoride of silicon. The metals present are found in the residue as sulphates (mixed with sulphate of calcium, if fluor spar has been used).

By fusion with carbonate of potassium and sodium. Applicable to all silicates. The silicate is fused with four parts of the carbonate of potassium and sodium until the evolution of carbonic acid has ceased, and the fused mass is then boiled with water, mixed with hydrochloric acid to strongly acid reaction, evaporated to dryness, and heated at a temperature rather above 100° until no more acid fumes escape, lumps being broken up with a pestle so that the whole may be completely dried. The powder is then moistened with hydrochloric acid, heated with water, and filtered. The filtrate contains the metals, and the residue contains all the silica.

By fusion with hydrate of barium. Applicable to all silicates. The silicate is fused with four parts of hydrate of barium, and the fused mass is treated as just mentioned.

Sulphuric Acid.

DRY REACTION.

1. Upon fusing a sulphate with *carbonate of sodium on charcoal* in the inner flame of the blowpipe, sulphide of sodium is formed. The fused mass will now evolve sulphuretted hydrogen if treated with an acid; or if placed upon a clean silver coin and moistened with acid, it will produce a black stain of sulphide of silver.

WET REACTIONS.

2. *Chloride of barium* produces a finely-divided, heavy

white precipitate of sulphate of barium ($BaSO_4$), which is insoluble in dilute hydrochloric and nitric acids. In the case of very dilute solutions, the mixture should be allowed to stand several hours; the trace of precipitate formed will then be found at the bottom of the vessel. Concentrated acids and concentrated solutions of many salts impair the delicacy of the reaction.

3. *Acetate of lead* produces a heavy white precipitate of sulphate of lead ($PbSO_4$), which is sparingly soluble in dilute nitric acid, but dissolves completely in hot strong hydrochloric acid.

4. *To detect free sulphuric acid* the fluid is mixed with a very little cane sugar and evaporated to dryness at 100°. A black residue will remain, or in the case of mere traces a blackish-green residue. No other free acid decomposes cane sugar in this way.

5. Insoluble sulphates, when fused with alkaline carbonates, give soluble alkaline sulphates.

GENERAL ANALYSIS.

GENERAL REMARKS.

Where there is reason to believe that a substance given for analysis is not mixed, and where it *exactly* resembles any common element, it may be examined as follows, according to *a* or *b*.

a. If the substance exactly resembles a certain non-metal, such as sulphur, iodine, carbon, &c., an examination of its properties—its behaviour under ignition, under treatment with solvents, &c.—will suffice to identify it. Thus if the substance looks like sulphur, it should be heated on a piece of porcelain; if it melts, burns with a blue flame, emits a smell of sulphurous acid, and leaves no residue, it may be considered to be sulphur. Thus, again, if the substance looks like blacklead, marks paper in the same way, and will not burn nor dissolve in acids, it may be considered to be blacklead.

b. If the substance exactly resembles a certain metal,* such as lead, copper, iron, &c., it should be divided finely if possible (by filing, cutting, or pounding), and heated with a mixture of equal parts of strong nitric acid and water till all action has finished. If a white powder is formed which will not dissolve even on dilution with water, this consists of oxide of tin or oxide of antimony, and indicates the presence of one of these metals; they are so different that you cannot be in doubt which the substance actually consists of. If on treating with water a clear solution is obtained, it should be examined according to p. 99, *et seq.*

But in general whatever the substance may look like, it should be examined by the prescribed course; indeed this is the only safe way of analysing it.

The study of analysis proper is usually divided into two

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* The student should be cautioned against mistaking alloys for simple metals. It may also be mentioned that certain metallic sulphides, such as galena, stibnite, iron pyrites, and copper pyrites, look like metals, but then they do not exactly resemble any individual metal.

parts, viz., the Analysis of Simple Substances (*i.e.*, oxides, acids, simple salts) and the Analysis of Mixed Substances. In going through the first part the student has substances given him to analyse in which he is not expected to find more than one metal and one acid. The mixtures given for analysis in the second part may contain any number of simple substances, and in analysing them the student is expected to look for every metal and every acid, and either to find it or prove its absence.

The chief object of the first part is to prepare the way for the second part.

The Preliminary Examination, Examination of Solubility, and Preparation of the Solution, are nearly the same in both parts.

PRELIMINARY (DRY) EXAMINATION.

Heat the substance in an ignition-tube before the blowpipe.

The substance sublimes.

1. Heat the substance with potash in a test-tube, and smell the vapor. If ammonia is given off, **ammonium** is indicated.
2. Heat the substance with dry carbonate of sodium in an ignition-tube. If globules of mercury sublime, **mercury** is indicated. If a steel-grey mirror is formed, **arsenic** is indicated.

Yellow drops of sulphur sublime. **Sulphur** is indicated.

Heat the substance with carbonate of sodium on charcoal in the inner blowpipe flame.

Metallic globules indicate **silver** or **lead** if malleable, **antimony** or **bismuth** if brittle.

A garlic odor indicates **arsenic.**

Deflagration indicates a **nitrate** or **chlorate.**

If the substance is colored, heat it in the borax bead in the outer blowpipe flame.

The cold bead is blue. **Cobalt** or **copper** is indicated.
green. **Chromium** is indicated.
dark-yellow or bottle-green. **Iron** is indicated.
reddish-brown. **Nickel** is indicated.
amethyst-red. **Manganese** is indicated.

Dip a clean platinum wire in strong hydrochloric acid, then in the substance, and hold it in the Bunsen flame.

The flame is colored azure. **Copper** is indicated.
green. **Copper** or **boracic acid** is indicated.
yellowish-green. **Barium** is indicated.
crimson. **Strontium** is indicated.
red. **Calcium** is indicated.
yellow. **Sodium** is indicated.
violet. **Potassium** is indicated.

EXAMINATION OF SOLUBILITY.

The solvents used are water, hydrochloric acid, nitric acid, and aqua regia. In this examination very small quantities of the substance are taken.

In the case of a simple substance, the powdered body is first boiled with water. If it will not dissolve in a moderate proportion, it should be considered to be insoluble in water, and very finely powdered before proceeding. Separate portions of the *fine* powder are then heated with dilute hydrochloric acid, strong hydrochloric acid, dilute nitric acid, strong nitric acid, and aqua regia (in this order), till a solvent is found or the substance is proved to be insoluble. Whenever a simple substance will not dissolve in a moderate proportion of any fluid, it is considered to be insoluble in that fluid. In heating a strong acid with a substance, the acid should not be boiled violently, as such boiling wastes the acid.

In the case of a mixed substance, if it cannot be dissolved completely with the aid of the above solvents, some of the constituents may be soluble. Whether this is so or not may be ascertained by filtering off the residue after the substance has been heated with a solvent, and evaporating a drop of the filtrate on a piece of porcelain.

Certain bodies show a peculiar deportment in this examination; thus, litharge, when boiled with strong nitric acid, is converted into nitrate of lead, which is insoluble in strong nitric acid, but will dissolve on addition of water. Thus, again, sulphide of antimony, when heated with strong nitric acid, is converted into white oxide, which does not dissolve in the nitric acid, but will dissolve immediately on the addition of strong hydrochloric acid.

If on heating a substance with acids it dissolves with exception of sulphur or gelatinous silica, the substance is considered to be soluble, the mixture of fluid and precipitate being treated as mentioned below.

PREPARATION OF THE SOLUTION.

In the case of a simple substance, dissolve it in the proper solvent.

In the case of a mixed substance, if any part is soluble, dissolve what you can and wash the residue, adding the first washings to the solution. (The residue is examined as an insoluble substance.)

If sulphur separates, the rest being dissolved, dilute and filter, wash the residue a little and heat it on a piece of porcelain, when it should burn with a smell of sulphurous acid; it may probably leave a residue, as it is liable to have some of the substance enclosed in it. If gelatinous silica separates, the rest being dissolved, evaporate the whole (with excess of acid) to dryness, and take up again with hydrochloric acid and water; the silica will be left undissolved.

If needle-shaped crystals are deposited from an acid solution, they probably consist of chloride of lead; they should be filtered off, washed, and examined at once. They should be soluble in boiling water, and one portion of the solution should give a yellow precipitate with bichromate of potassium, while another portion should give a white curdy precipitate with nitrate of silver insoluble in nitric acid.

If on diluting an acid solution a white precipitate forms, antimony or bismuth is indicated. The precipitate may at once be redissolved by the addition of hydrochloric acid if necessary.

When nitric acid is used for dissolving the substance, mercury, tin and iron, if present, will be converted into the dyad, tetrad and triad states respectively, and arsenic, if present, will be converted into arsenic acid; consequently if any of these metals are found, the original substance must be examined to see in what state they are actually present.

ANALYSIS OF SIMPLE SUBSTANCES.

In which the student is not expected to find more than one metal and one acid.

SOLUBLE SUBSTANCES.

DETECTION OF THE METAL.

The indications afforded by the dry examination are in certain cases so conclusive as to leave no room for doubt concerning which metal is present. However, as the object of this part is practice, the student should in all cases proceed to the wet examination as follows.

As soon as one metal is found, the student should stop and go on to the Detection of the Acid, unless the metal is arsenic or chromium. If arsenic is found, it was probably present as an acid, and another metal must be looked for. If chromium is found, and chromic acid is present in the original solution, another metal must also be looked for.

Treat some of the solution as follows, reserving the rest for any tests that may have to be applied to it directly in the subsequent course.

1. Add hydrochloric acid in good excess (if not already used in dissolving). If a precipitate is formed, take a small portion of the mixed precipitate and fluid and add to it ammonia in excess. The precipitate

redissolves. Indication of **silver.**

turns black. Indication of **monad mercury**. Confirm by adding stannous chloride to the original solution.

appears unaltered. Indication of **lead.** Confirm by throwing the rest of the mixed precipitate and fluid onto a filter, and boiling the precipitate with water, when it should redissolve.

2. To the fluid treated with hydrochloric acid, add sulphuretted hydrogen water* until it distinctly smells after shaking, and heat. If a white precipitate is formed, proceed as if there were no precipitate, as it consists of sulphur separated from the sulphuretted hydrogen by triad iron or chromic acid, &c. When chromic acid is present, besides sulphur being precipitated, the solution which was originally red changes to green. If a colored precipitate is formed, and it is

- *yellow.* This consists of sulphide of cadmium, sulphide of arsenic or stannic sulphide. Mix a portion of the fluid containing the precipitate with excess of ammonia, add sulphide of ammonium, and heat. The precipitate
  - *remains undissolved.* It is sulphide of **cadmium.**
  - *dissolves.* It is sulphide of **arsenic** or sulphide of **tetrad tin.** Remembering the results of the preliminary examination you will not be in doubt which.
- *orange colored.* It is sulphide of **antimony.** Confirm by treating the original solution with zinc and hydrochloric acid in a platinum crucible lid.
- *dark colored.* It is sulphide of lead, mercuric sulphide, sulphide of bismuth, sulphide of copper, or stannous sulphide. To the original solution add
  - *sulphuric acid.* A white precipitate indicates **lead.** Confirm by testing the original solution with bichromate of potassium.
  - *potash.* A yellow precipitate indicates **dyad mercury.**
  - *ammonia in excess.*
    - *A white precipitate* is formed. Filter it off, dissolve it in a drop or two of hydrochloric acid on a watch-glass, and then dilute. A precipitate indicates **bismuth.**
    - *A blue color* indicates **copper.**
  - *mercuric chloride.* A white precipitate indicates **dyad tin.**

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* If arsenic was found in the preliminary examination it may be present here as arsenic acid, when it would not be precipitated readily by sulphuretted hydrogen. In this case, therefore, the solution should be boiled with acid sulphite of ammonium, and the excess of sulphurous acid driven off, before sulphuretted hydrogen is added.

3. To the fluid mixed with hydrosulphuric acid, add chloride of ammonium, excess of ammonia, and sulphide of ammonium. The precipitate is

black. It consists of sulphide of nickel, sulphide of cobalt, or sulphide of iron. To the original solution add potash. The precipitate is

light green, and does not change color on exposure to the air. **Nickel** is indicated. This is confirmed by the results of the dry examination.

blue. **Cobalt** is indicated. Found already in the dry examination.

dirty green, and soon turns brown on exposure to the air. Indication of **dyad iron.** Confirm by adding ferricyanide of potassium to the original solution.

brown. Indication of **triad iron.** (The solution should have given a precipitate of sulphur on the previous treatment with hydrosulphuric acid.) Confirm by adding sulphocyanide of potassium to the original solution.

*not black.** It is sulphide of manganese, sulphide of zinc, hydrate of chromium, hydrate of aluminium, silicic acid, or a silicate, phosphate, borate or fluoride of barium, strontium, calcium or magnesium.† **Manganese** will have been indicated in the dry examination; its presence may be proved by fusing some of the precipitate in a platinum loop with carbonate of sodium and nitrate of potassium. **Chromium** will have been found already in the dry examination. If neither of these two metals are present, evaporate a portion of the original solution to dryness with hydrochloric acid, moisten the residue with hydrochloric acid, and add water. If a residue remains, it is **silica;** filter it off, and test the filtrate for the metal (barium, strontium, calcium, magnesium, potassium, or sodium) as directed **4** *et seq.*, p. 103. If silica is absent, add tartaric acid to a portion of the original solution, and then ammonia in excess.

No permanent precipitate is formed. Indication of

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* If the precipitate is pure flesh-color, manganese is indicated; if pure bluish-green, chromium is indicated; if pure white, one of the other bodies is indicated. But traces of iron, which are often present, will often darken these colors.

† If the body was soluble in water, these salts are not present.

zinc or aluminium. Mix the original solution with excess of potash, and divide the clear solution in half.

1. Add hydrosulphuric acid. A precipitate indicates **zinc.**
2. Add chloride of ammonium and heat. A precipitate indicates **aluminium.**

A permanent precipitate is formed. Indication of a phosphate, borate, or fluoride of barium, strontium, calcium, or magnesium. Proceed as follows:—

1. To the original hydrochloric acid solution add ammonia, till a precipitate forms, then acetic acid till this is redissolved, lastly acetate of sodium and a drop of ferric chloride; the formation of a white flocculent precipitate indicates **phosphoric acid.** If phosphoric acid is present, add now some more ferric chloride, until the fluid is distinctly red, boil, filter boiling, and test the filtrate, which is now free from phosphoric acid, for the alkaline earthy metal, as directed **4** *et seq.*, p. 103, after having separated by ammonia any iron which may have dissolved.
2. **Boracic acid** is tested for by nearly neutralizing the hydrochloric acid solution, moistening turmeric paper with it, and drying the paper. If boracic is present, the metal is found by boiling the original substance with carbonate of sodium, filtering off the carbonate produced, washing it, dissolving it in the least quantity of hydrochloric acid, evaporating to dryness, dissolving the residue in water, and testing as directed **4** *et seq.*, p. 103.
3. **Hydrofluoric acid** is detected by treating a portion of the original substance with strong sulphuric acid, and etching glass with the gas evolved. To discover the metal, the residue is ignited, to drive off all the hydrofluoric acid, and the excess of sulphuric acid, powdered and fused with carbonate of potassium and sodium. The fused mass is poured out onto a cold stone, powdered, boiled with water, and filtered. The residue on the filter is washed well with water, dissolved in hydrochloric acid, and tested according to **4** *et seq.*

4. To the fluid mixed with chloride of ammonium, ammonia and sulphide of ammonium, add carbonate of ammonium, and heat gently. If a precipitate is formed, filter it off, wash it, and dissolve in the least quantity of warm hydrochloric acid by pouring the acid on the filter. To a portion of the solution add sulphate of calcium.

A precipitate forms immediately. **Barium** is indicated.

A precipitate forms after some time. **Strontium** is indicated. The substance will have tinged the Bunsen flame crimson in the preliminary examination.

No turbidity occurs even after ten minutes' standing. **Calcium** is indicated. Confirm by adding ammonia in excess and oxalate of ammonium to another portion of the dissolved precipitate.

5. To the fluid mixed with carbonate of ammonium add phosphate of sodium and stir well.

A crystalline precipitate is formed. **Magnesium** is indicated.

No precipitate is formed. Potassium, sodium, or ammonium is probably present. Dissolve some of the original substance in the least quantity of water in a watch-glass, add a few drops of hydrochloric acid and a few drops of chloride of platinum to the solution, and stir well.*

A yellow precipitate is formed. Indication of **potassium** or **ammonium.** Which is actually present cannot be doubted if the result of the first preliminary experiment is remembered, since potassium compounds are fixed and ammonium compounds volatile.

No precipitate is formed. **Sodium** is indicated. The substance should have given a yellow color in the Bunsen flame in the preliminary examination.

DETECTION OF THE ACID.

Nitric acid or chloric acid will have been already indicated

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* If a dark color (from iodine) is produced on the addition of the platinum solution, the experiment should be repeated in the same manner, with the substitution of a strong solution of tartaric acid for the hydrochloric acid and chloride of platinum. A white crystalline precipitate will indicate potassium or ammonium.

by the deflagration on charcoal in the preliminary examination. The presence of **nitric acid** may be proved by dissolving the substance in water, adding solution of ferrous sulphate, and then holding the tube obliquely and pouring strong sulphuric acid down to the bottom. The presence of **chloric acid** may be proved by adding sulphuric acid to the aqueous solution of the substance and then indigo solution and sulphurous acid.

Chromic acid will have been indicated already by the red color of the solution on addition of hydrochloric acid, and by its turning green on the subsequent addition of hydrosulphuric acid.

Silicic acid will have been found already.

If neither of these acids is present, refer to the Table of Solubility at the end of the book, consider which acids may be present, and look for these.

1. Hydrosulphuric and carbonic acids may be detected by heating the dry powdered substance with dilute hydrochloric acid in a test-tube.

If **hydrosulphuric acid** is present it will be given off, and may be recognised by its smell, or by its turning paper wetted with acetate of lead black.

If **carbonic acid** is present, carbonic anhydride will be given off with brisk effervescence; the gas may be recognised by its want of odor, and by its giving a precipitate with lime-water after being poured off into a fresh test-tube.

2. Hydrochloric, hydrobromic, and hydriodic acids are detected as follows. If the substance is soluble in water, dissolve in water, acidify with nitric acid, and to a portion of the solution add nitrate of silver. If the substance is insoluble in water, boil it (the dry substance) with solution of carbonate of sodium, filter off the residue, acidify the filtrate with nitric acid, and to a portion add nitrate of silver. In either case, hydrochloric acid will give a white precipitate; hydrobromic or hydriodic acid a yellowish white precipitate.

To settle which acid is present, to the rest of the solution acidified with nitric acid add a drop or two of chloride of soda, a few drops of bisulphide of carbon, and shake. If the bisulphide remains colorless, **hydrochloric acid** is present; if it is colored yellow, **hydrobromic acid** is present; if it is colored purple, **hydriodic acid** is present.

3. **Hydrofluoric acid** is detected by warming the dry powdered substance in a platinum crucible with strong sulphuric acid, the crucible being covered with a piece of glass.

4. **Boracic acid** is detected by boiling the substance with carbonate of sodium, filtering, boiling the filtrate with slight excess of hydrochloric acid,* dipping a piece of turmeric paper in the fluid, and drying it.

5. **Phosphoric acid** is detected by adding the molybdate of ammonium solution to the original solution.

6. **Sulphuric acid** is detected by adding hydrochloric acid to the original solution and then chloride of barium. (Make sure that the precipitate is not soluble in water, for if the solution is very acid the chloride of barium itself will be thrown down.)

If no acid is present, and the substance has the properties of an oxide of the metal found, you may conclude that it is such.

INSOLUBLE SUBSTANCES.

COMPLETE ANALYSIS.

The following is a list of the substances which we take into consideration here :—

Silver Chloride.
Lead Chloride.
Sulphate.
Barium Sulphate.
Strontium Sulphate.
Calcium Fluoride.
Sulphate.
Silica.
Silicates.

Heat the substance in a bead of microcosmic salt.

If the substance does not dissolve, it is silica or a silicate.

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* If the metal present belongs to the fifth group, it will suffice to add a slight excess of hydrochloric acid to the aqueous solution of the substance before testing with turmeric paper.

Powder it very finely, and fuse it with six parts of carbonate of potassium and sodium, pour out the fused mass onto a stone, powder it, add water and excess of hydrochloric acid, evaporate to dryness on a water-bath, breaking up the lumps with a pestle so that the whole may be thoroughly dried, moisten with hydrochloric acid, add water, heat and filter. The residue is **silica.** Examine the filtrate for the **metal** beginning at p. 99.

If the substance dissolves, add to the original substance on a watch-glass a drop of sulphide of ammonium.

If the substance turns black, silver or lead is present. Fuse the original substance with six parts of carbonate of potassium and sodium, pour the fused mass onto a stone, powder it finely, boil with water, and filter.

The residue consists of **silver** or oxide of **lead.** Wash it, dissolve in nitric acid, and examine the solution according to **1,** p. 99.

The filtrate contains the hydrochloric or sulphuric acid in the form of alkaline salt, together with the excess of carbonate of potassium and sodium. Acidify with nitric acid and boil, divide into two parts.

1. Add nitrate of silver and shake. A curdy precipitate indicates **hydrochloric acid.**
2. Add chloride of barium. A precipitate indicates **sulphuric acid.**

If the substance remains white, test for sulphuric acid by heating the finely powdered substance with carbonate of sodium on charcoal.

If sulphuric acid is present. Fuse the finely powdered substance with six parts of carbonate of potassium and sodium, pour out onto a stone, powder finely, boil with water, and filter. *The filtrate* contains the **sulphuric acid** as an alkaline salt; on acidifying it with hydrochloric acid and adding chloride of barium, it will be precipitated. *The residue* consists of carbonate of **barium, strontium,** or **calcium;** it should be well washed, dissolved in the least quantity of hydrochloric acid, and tested according to **4,** p. 103.

If sulphuric acid is absent, mix the finely powdered substance with strong sulphuric acid in a platinum crucible, and apply the etching-test for **hydrofluoric acid.** If fluorine is found, heat the crucible till all the hydrofluoric acid and the excess of sulphuric acid are driven off, boil the residue with hydrochloric acid, filter, add excess of ammonia to the filtrate, and then oxalate of ammonium to test for **calcium.**

ANALYSIS OF MIXED SUBSTANCES.

In which all the metals and all the acids may be present.

SOLUBLE SUBSTANCES.

DETECTION OF METALS.

In the following course, if any group precipitate should be very small, it will be useless to examine it for all the members in the regular way, as even if they were present they could not be found. Under these circumstances the precipitate should be examined for one or two metals which are suspected, or in particular cases a large quantity of the original solution should be taken, and enough of the precipitate prepared for a complete examination.

1. Precipitation of the Metals of Group I. by Hydrochloric Acid, and Examination of the Precipitate.

To the solution of the substance add a few drops of hydrochloric acid. If a precipitate is produced, add more acid till the precipitation is complete, and then a few drops more (to dissolve basic chlorides of antimony and bismuth). Filter and examine the filtrate by **2** *a*.

The precipitate may contain silver, mercurous and lead chlorides.* Wash it twice with cold water to remove foreign substances, and then wash three times on the filter with hot water.

f_1† Add sulphuric acid. A white precipitate indicates **lead.**

p_1† *on the filter.* Add ammonia.

f_2 (If it is not quite clear, take no notice, as the basic lead-salt which causes the turbidity is soluble in

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* It will, of course, be white. If the solution was an alkaline one, the following substances might be thrown down here,—gelatinous silica, sulphur, chloride of silver, chloride of lead, sulphate of lead, and stannic, antimony, and arsenious sulphides. If silica or sulphur are suspected, proceed as directed p. 98, Preparation of the Solution, 3rd par. (sulphur will be difficult to filter off); otherwise treat the precipitate as a fresh substance.

† f is used for *filtrate* or *solution*,
p is used for *precipitate* or *residue*.

nitric acid.) Add excess of nitric acid. A precipitate indicates **silver.**

p_2 If it is black or grey, **monad mercury** is indicated.

2 *a.* PRECIPITATION OF THE METALS OF GROUP II. BY HYDROSULPHURIC ACID, AND SEPARATION OF THE PRECIPITATE INTO TWO DIVISIONS.

If arsenic was found in the Preliminary Examination, before proceeding, boil the filtrate from the hydrochloric acid precipitate with acid sulphite of ammonium, adding more hydrochloric acid if necessary, and boiling till the excess of sulphurous acid is driven off.

In order to find out whether hydrosulphuric acid produces a precipitate or not, and thus to be enabled to save the trouble of passing the gas if it is unnecessary, take a small quantity of the filtrate from the hydrochloric acid precipitate (which contains already a moderate excess of hydrochloric acid), and add hydrosulphuric acid solution in excess—*i. e.*, till on shaking the test-tube with the thumb on the top, the contents smell distinctly of the reagent. If only sulphur is precipitated, take no notice of the precipitate here; if a colored precipitate is formed, proceed as follows.

Pass hydrosulphuric acid gas through the rest of the filtrate from the hydrochloric acid precipitate, diluting it if it is very acid. When the precipitate has completely formed, filter and examine the filtrate by **3** *a*, p. 112. The precipitate may contain (1) lead, mercuric, bismuth, copper and cadmium sulphides; also (2) tin, antimony, and arsenious sulphides. Wash it well.

Before we treat the whole precipitate with sulphide of ammonium, to separate the first division of metals from the second division, it may save trouble to see if this treatment is necessary—*i. e.*, if the precipitate really contains members of both divisions. To this end, warm a small quantity with ten or twenty drops of yellow sulphide of ammonium.*

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* If the preliminary examination has shown that copper is present and mercury absent, when sulphide of ammonium is directed to be used in the examination of this group, use sulphide of sodium instead, and boil. In sulphide of ammonium sulphide of copper is a little soluble. In sulphide of sodium the copper sulphide is insoluble, but the mercuric sulphide is soluble.

If the precipitate completely dissolves. **The members of the first division are absent.** Treat the rest of the precipitate as directed **2** *c*.

If the precipitate does not completely dissolve. Filter off the residue, and add hydrochloric acid to the filtrate.

If a white precipitate is formed. It consists merely of sulphur from the sulphide of ammonium. **The members of the second division are absent.** Treat the rest of the precipitate as directed **2** *b*.

If a colored precipitate is formed. Members of both divisions are present.

If members of both divisions are present, warm the rest of the hydrosulphuric acid precipitate with yellow sulphide of ammonium, and filter.

p. Examine according to **2** *b*.

f. Add hydrochloric acid in excess to reprecipitate the sulphides, filter, and treat the precipitate according to **2** *c*.

2 *b*. Examination for Metals of Gr. II. Div. I., and Lead.

The precipitate may contain lead, mercuric, bismuth, copper and cadmium sulphides. Wash it thoroughly, boil with strong nitric acid, dilute and filter.

p_1. may contain sulphur (yellow globules), mercuric sulphide (black), lead sulphate (white), and stannic oxide* (white). Divide it into two parts.

1. Dissolve in hydrochloric acid with the addition of a little chlorate of potassium, and add stannous chloride. A grey or white precipitate indicates **dyad mercury.**

2. Fuse with cyanide of potassium and carbonate of sodium. If metallic globules are formed, wash and treat them with nitric acid.

The solution. Add sulphuric acid. A precipitate indicates **lead.**

The residue may be metastannic acid. Wash it well and boil with strong hydrochloric acid for a few minutes, pour off the acid, add water (see *Note*, p. 36), and then hydrosulphuric acid. A yellow precipitate indicates **tin.**

f_1 may contain lead, bismuth, copper and cadmium. To a

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* The separation of tin by sulphide of ammonium is often incomplete.

small portion add sulphuretted hydrogen to see if there are any metals in solution. If a precipitate is produced, to the rest of the solution add sulphuric acid, evaporate on the water-bath till the nitric acid is expelled, take up with water, and filter.

p_2 is sulphate of **lead.**

f_2 Add excess of ammonia, warm and filter.

p_3 Wash, dissolve in a watch glass in the least hydrochloric acid, and add water. A milkiness indicates **bismuth.**

f_3 If it is blue, **copper** is indicated. (To test for very small quantities of copper, evaporate to dryness and add a drop or two of ammonia.) *If copper is absent,* test for cadmium at once by adding hydrosulphuric acid. *If copper is present,* precipitate with sulphuretted hydrogen, wash the precipitate, boil it with dilute sulphuric acid, filter, and add hydrosulphuric acid to the filtrate. A yellow precipitate in either case indicates **cadmium.**

2 *c.* EXAMINATION FOR METALS OF GR. II. DIV. II.

The precipitate may contain stannous, stannic, antimonious and arsenious sulphides. Wash it thoroughly.

First ignite a small quantity on a piece of porcelain.

If it is completely volatile, **only arsenic is present.** Dissolve another portion of the precipitate in a few drops of strong nitric acid on a watch-glass, dilute, filter off the sulphur, add nitrate of silver, and then diluted ammonia carefully so as not to mix the fluids. A red cloud indicates arsenic.

If a residue is left, **all the metals must be looked for;** proceed as follows.

Dry the rest of the precipitate on the filter, and divide it into two portions.

1. Boil with strong hydrochloric acid (which will dissolve the tin and antimony), dilute a little, filter into a platinum crucible lid, and add a little piece of zinc plate to the filtrate. A black stain indicates **antimony.** Take out the zinc, wash off any adherent powder back into the platinum crucible lid,

pour away the solution, boil the residue with strong hydrochloric acid, dilute a little, and filter; to the filtrate add mercuric chloride. A white or grey precipitate indicates **tin.**

2. Fuse with cyanide of potassium and carbonate of sodium in an ignition-tube. A mirror indicates **arsenic.**

3 *a*. PRECIPITATION OF THE METALS OF GROUP III. AND SILICATES, PHOSPHATES, BORATES, AND FLUORIDES OF BARIUM, STRONTIUM, CALCIUM, AND MAGNESIUM, AND ALSO SILICA, BY CHLORIDE OF AMMONIUM, AMMONIA, AND SULPHIDE OF AMMONIUM. AND PRELIMINARY DETERMINATION OF THE NATURE OF THE PRECIPITATE.

If the substance was soluble in water. **Salts and silica* are absent.** A portion of the filtrate from the hydrosulphuric acid precipitate is boiled to drive off the excess of the gas, mixed with a few drops of strong nitric acid and boiled again, then mixed with chloride of ammonium and ammonia till the reaction is just alkaline. If the fluid remains clear, iron, chromium, and aluminium are also absent.

If the substance was insoluble in water. Salts and silica may be present. A portion of the filtrate from the hydrosulphuric acid precipitate is boiled to expel excess of the gas, mixed with a few drops of strong nitric acid and boiled again, then mixed with chloride of ammonium and ammonia, till the reaction is just alkaline. If the fluid remains clear, **salts and silica,** also iron, chromium, and aluminium, **are absent.**

To the rest of the filtrate from the hydrosulphuric acid precipitate add chloride of ammonium, ammonia just to alkaline reaction, and then a drop or two of sulphide of ammonium; if a precipitate is formed, continue adding sulphide of ammonium, till after brisk shaking the smell of it is distinctly recognisable. Filter, and examine the filtrate,† as directed **4** *a*. The precipitate is examined as directed **3** *c* or **3** *b*, according as salts and silica have to be looked for or not.‡

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* If the original dry substance was very alkaline, silica may be present.

† If it is blackish, this is caused by nickel, whose sulphide is slightly soluble in sulphide of ammonium. Take no further notice.

‡ Alkaline earths may be precipitated in combination with chromium. In very careful analyses the second method should be always adopted.

3 *b*. EXAMINATION OF THE SULPHIDE OF AMMONIUM PRECIPITATE WHEN SALTS AND SILICA ARE ABSENT.

The precipitate may contain nickel, cobalt, iron,* manganese, zinc (as sulphides), chromium, aluminium (as hydrates).

Wash it with water containing a little sulphide of ammonium, remove it from the filter, treat with cold dilute hydrochloric acid, and filter.

p_1 (*black*) may contain nickel and cobalt sulphides. Wash and test it in the borax bead. *A reddish-brown bead* indicates the presence of **nickel,** and the absence of cobalt. *A blue bead* indicates the presence of **cobalt.** In the latter case, and also if the result is indefinite, proceed as follows:—Dry the filter containing the precipitate, incinerate it (see p. 21), dissolve the ash in a few drops of aqua regia, dilute, filter, nearly neutralize with potash, and add nitrite of potassium and then acetic acid to strongly acid reaction, allow to stand a long time, and filter.

A yellow precipitate indicates **cobalt.**

The filtrate. Add potash; if a precipitate is produced, filter it off and test it for **nickel** in the borax bead.

f_1 may contain iron, chromium, aluminium,† manganese, and zinc. Add a small quantity of strong nitric acid, and boil, nearly neutralize with potash; when cold, add excess of carbonate of barium, allow to stand in the cold and filter.

p_2 may contain iron, chromium, and aluminium. Wash well and divide it into three parts.

1. Dissolve in hydrochloric acid, and add sulphocyanide of potassium. A red color indicates **iron.**
2. Boil with chloride of soda, and filter. A yellow filtrate indicates **chromium.** If the filtrate is pink (from manganese not washed out of the carbonate of barium precipitate), add alcohol, boil and filter off the brown oxide of manganese formed; if chromic acid is present, its color will then be visible.
3. Boil with potash, and filter, add excess of chloride

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* These first three metals cannot be present if the precipitate has a pure white color.

† If the preliminary examination of the sulphide of ammonium precipitate has shown the absence of these first three metals, you may pass on at once to examine for manganese and zinc.

of ammonium and boil again. A flocculent precipitate indicates **aluminium.**[*]

f_2 may contain manganese and zinc. First, to get rid of the barium, add sulphuric acid and filter through charcoal. Then evaporate to a small bulk, add potash in excess, and filter.

p_3 may contain manganese, also traces of nickel and cobalt originally dissolved by the hydrochloric acid. Test for **manganese** with the carbonate of sodium bead.

f_3 Add hydrosulphuric acid. A white precipitate indicates **zinc.**

3 *c*. EXAMINATION OF THE SULPHIDE OF AMMONIUM PRECIPITATE WHEN SALTS AND SILICA MAY BE PRESENT.[†]

The precipitate may contain nickel, cobalt, iron,[‡] manganese, zinc (as sulphides), chromium, aluminium (as hydrates), barium, strontium, calcium, magnesium (as silicates and other salts), and silica.

Wash it with water containing a little sulphide of ammonium, remove it from the filter, treat with cold dilute hydrochloric acid, and filter.

p_1 may contain sulphide of nickel (black), sulphide of cobalt (black), and silica (white). Wash and test it with a bead of microcosmic salt. *If a skeleton,* **silica** is present; *if a yellow bead,* **nickel** is present and cobalt absent; *if a blue bead,* **cobalt** is present. If cobalt is present, and also if the bead has an indefinite color, proceed as follows. Dry the filter containing the precipitate, incinerate it (see p. 21), dissolve the ash in a few drops of aqua regia, dilute, filter, nearly neutralize with potash, add nitrite of potassium, and then acetic acid to strongly acid reaction, allow to stand for a long time, and filter.

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[*] The potash used generally itself gives a precipitate with chloride of ammonium; therefore, if a precipitate is obtained here, it is well to compare its bulk with the bulk of a precipitate obtained by boiling excess of chloride of ammonium with a quantity of potash equal to that used before. For delicate analyses of course pure potash (or soda, see p. 12) must be used.

[†] With the exception of silica the acids are not looked for here, as they will be found in the regular examination for acids.

[‡] The first three metals cannot be present if the precipitate has a pure white color.

A yellow precipitate indicates **cobalt.**

The filtrate. Add potash; if a precipitate is produced, filter it off and test it for **nickel** in the borax bead.

f_1 Boil to expel hydrosulphuric acid, filter if necessary, and divide into two parts, the first much smaller than the second.

1. Add dilute sulphuric acid, and filter.

The precipitate may contain **barium, strontium,** and possibly also calcium. Wash and examine it by the spectroscope, or fuse it with carbonate of potassium and sodium, treat with water, wash, and examine as directed **4** *a*.

The filtrate. Mix with three volumes of alcohol. A precipitate consists of sulphate of **calcium.** Dissolve it in water, and test the solution with oxalate of ammonium.

2. Add some strong nitric acid, and boil. Test a small portion with sulphocyanide of potassium for **iron.** To the rest add ferric chloride (to separate phosphoric and silicic acids) till a drop on a watch-glass gives a yellowish precipitate with ammonia, evaporate on a water-bath to a small bulk, add water, nearly neutralize with potash, then add carbonate of barium in excess, and allow to stand in the cold. Filter.

p_2 Wash well and divide into two parts.

1. Boil with chloride of soda, and filter. A yellow filtrate indicates **chromium.**

2. Boil with potash, and filter. To the filtrate add excess of chloride of ammonium, and boil; a precipitate may contain alumina and silica. Test for **silica** by the microcosmic bead. If it is present, ignite the rest of the precipitate on a platinum crucible lid, add acid sulphate of potassium, fuse, and treat with hydrochloric acid. The silica is left behind, and alumina, if present, is dissolved. Add ammonia to the solution; a precipitate indicates **aluminium.**

f_2 Add a few drops of hydrochloric acid, boil to expel carbonic acid, and add ammonia and sulphide of ammonium. Filter.

p_3 Wash with water containing a little sulphide of ammonium, treat with acetic acid, and filter.

p_4 may contain zinc and traces of nickel and cobalt. Wash it, treat with dilute hydrochloric acid, and filter if necessary. Treat the filtrate with nitric acid, evaporate to a small bulk, add potash, boil, and filter if necessary. Test the filtrate with sulphide of ammonium for **zinc.** If a black residue is left on the treatment with hydrochloric acid, or if a residue insoluble in potash is left, test it for nickel and cobalt.

f_4 Boil with potash, filter off the precipitate, and test it in the carbonate of sodium bead for **manganese.**

f_3 First remove the barium by adding sulphuric acid, boiling and filtering, then supersaturate with ammonia, add oxalate of ammonium, and filter off any oxalate of calcium which may be precipitated. Test the filtrate with phosphate of sodium for **magnesium.**

4 *a*. PRECIPITATION OF THE METALS OF GROUP IV. BY CARBONATE OF AMMONIUM (IN THE PRESENCE OF CHLORIDE OF AMMONIUM), AND EXAMINATION OF THE PRECIPITATE.

To the filtrate from the sulphide of ammonium precipitate, add carbonate of ammonium, warm gently, and filter. Examine the filtrate as directed **4** *b*.

The precipitate may contain barium, strontium, and calcium carbonates. Wash it, dissolve in the least quantity of hydrochloric acid, evaporate on the water-bath to dryness, and take up with a little water.

To a portion of the solution add sulphate of calcium, and allow to stand for some time.

No precipitate is formed. Absence of barium and strontium. Test the rest of the solution with oxalate of ammonium for **calcium.**

A precipitate is formed after some time. Absence of barium, presence of **strontium.** Examine for calcium as follows. Boil the rest of the solution with a strong solution of sulphate of ammonium for some time, adding more ammonia, if necessary, to keep the solution alkaline; filter off the undissolved sulphate of strontium, and add oxalate of ammonium. A precipitate indicates **calcium.**

A precipitate is formed immediately. Presence of **barium.** In order to test for strontium and calcium, proceed as follows:—Evaporate the rest of the solution to dryness, digest the residue with alcohol, using a pestle to bring the alcohol in contact with every particle of the residue, and filter off the undissolved chloride of barium. The filtrate contains the strontium and calcium and traces of barium; add sulphuric acid, and filter. Boil the precipitate with strong solution of sulphate of ammonium and a little ammonia for some time, and filter.

The residue consists of sulphate of **strontium** (and traces of sulphate of barium). Test it with the flame.

The filtrate. Dilute and test with oxalate of ammonium for **calcium.**

4 *b.* EXAMINATION OF THE FILTRATE FROM THE CARBONATE OF AMMONIUM PRECIPITATE FOR TRACES OF BARIUM AND CALCIUM, AND REMOVAL OF THE SAME.

To one portion add sulphate of ammonium, in order to test for barium; to another portion add oxalate of ammonium, and allow to stand, in order to test for calcium. If these metals are present, remove them from the rest of the filtrate by sulphate and oxalate of ammonium before passing on to **5** *a.*

EXAMINATION FOR METALS OF GROUP V.

5 *a.* To a portion of the fluid from which barium, strontium, and calcium have been separated, add phosphate of sodium, stir well, and allow to stand for a short time. A crystalline precipitate[*] indicates **magnesium.**

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[*] A slight flocculent precipitate of phosphate of aluminium sometimes separates here; on filtering it off it will be found to be soluble in potash, whereas phosphate of magnesium and ammonium is insoluble. The appearance of this precipitate is explained as follows. When aluminium is present in the original substance, and too large an excess of ammonia has been used with the sulphide of ammonium for the precipitation of Group III., a little hydrate of aluminium passes into the filtrate; this is not thrown down by the carbonate of ammonium, but on addition of phosphate of sodium, phosphate of aluminium is formed, which is far less soluble in ammonia than the hydrate of aluminium.

If magnesium is not present. Evaporate a small quantity of the rest of the solution on a platinum crucible lid, and ignite.

If no fixed residue is left. Potassium and sodium are absent.

If a fixed residue is left. Evaporate the rest of the solution to dryness, transfer to a piece of porcelain or a platinum crucible lid, and ignite till white fumes cease to be evolved. Dissolve the residue in the least quantity of water, and filter if necessary into a watch-glass. Pass on to **5** *b*.

If magnesium is present, it must be removed from the rest of the solution. Evaporate the latter to dryness, transfer to a platinum crucible lid, ignite till white fumes cease to be evolved, warm the residue with a little water, add baryta water (or milk of lime) till the reaction is strongly alkaline, boil, filter, mix the filtrate with a little ammonia and carbonate of ammonium, warm gently, filter, evaporate and ignite. Dissolve the residue in the least quantity of water, add a drop of hydrochloric acid, pour the solution into a watch-glass, and pass on to **5** *b*.

5 *b*. Having got rid of the ammonium and also of the magnesium if present, proceed to test for potassium and sodium as follows:—Dip a clean platinum wire into the solution, and hold it in the Bunsen flame; a yellow color indicates **sodium.** Then add to the solution a few drops of chloride of platinum, and stir; a yellow precipitate indicates **potassium.** If no precipitate is formed, evaporate to dryness at a gentle heat and add a few drops of water; the least trace of potassium will then be indicated by a heavy yellow insoluble powder.

If ammonium has not been found in the preliminary examination, put a small quantity of the original substance into a test-tube, add a little potash, and warm, with your thumb on the top of the tube. Then remove your thumb, and smell the contents of the tube. A smell of ammonia indicates **ammonium.**

SOLUBLE SUBSTANCES.

DETECTION OF ACIDS.

Warm some of the original substance with rather strong hydrochloric acid.

Effervescence with evolution of an odorless gas. Presence of **carbonic acid.** Confirm by pouring the gas in the test-tube into another test-tube containing a little lime-water, and shaking the latter.

Effervescence with evolution of sulphuretted hydrogen. Presence of **hydrosulphuric acid.** Test for **carbonic acid** by passing the gas into lime-water by the aid of a cork and a bent tube.

Dissolve the substance in its proper solvent, acidify with hydrochloric acid if not already acid, and divide into three portions.

1. Evaporate to dryness. An insoluble residue indicates **silicic acid.**
2. Add solution of molybdate of ammonium, and allow to stand in the cold. A yellow precipitate indicates **phosphoric acid.**
3. Add chloride of barium. A white precipitate indicates **sulphuric acid.** Make sure that the precipitate is not soluble in water.

To the original dry substance in a watch-glass add strong sulphuric acid. If the acid is colored yellow, and a smell of chlorine is evolved, **chloric acid** is indicated.

Test for **nitric acid** as follows :—Boil the original substance with a small quantity of water, filter (if the filtrate is alkaline add excess of hydrochloric acid), mix with solution of ferrous sulphate and pour strong sulphuric acid gently to the bottom of the tube.

For the following experiments prepare a solution as follows :

If only alkali metals are present, dissolve the original substance in water.

If other metals are present, boil the dry substance with solution of carbonate of sodium,* filter, boil the filtrate, and neutralize it with nitric acid.

Take separate portions of the solution prepared as above.

1. Acidify with hydrochloric acid, and add sulphate of copper. A black precipitate indicates **hydrosulphuric acid.**

2. Acidify with nitric acid, and if hydrosulphuric acid is present, boil the solution till it is expelled. Then add nitrate of silver.

   No precipitate is produced. Chlorine, bromine, and iodine are absent.

   A precipitate is produced. Take another portion of the solution, add large excess of hydrochloric acid, then a little bisulphide of carbon and a drop of chloride of soda, and shake. **Iodine,** if present, will be at once indicated by the color of the bisulphide of carbon. Add now more chloride of soda drop by drop, well shaking till the violet color disappears. If the violet color is replaced by a yellow color, **bromine** is present. If bromine and iodine are absent, of course you can assume that chlorine is present. If bromine or iodine is present, you must test for **chlorine** specially by distilling with bichromate of potassium and strong sulphuric acid.

3.† Acidify with acetic acid, and add acetate of lead. A yellow precipitate indicates **chromic acid.**

4. Acidify with acetic acid, and add chloride of calcium. A precipitate indicates **hydrofluoric acid.** Apply the etching test to the original substance.

5. Acidify slightly with hydrochloric acid, dip turmeric paper in the solution, and dry it. A brown stain indicates **boracic acid.**

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* In the case of substances insoluble in water, sometimes merely boiling with the solution is not sufficient to dissolve the acids, and fusion with the dry carbonate of sodium is necessary.

† You need not test for chromic acid if the solution is colorless.

INSOLUBLE SUBSTANCES.

COMPLETE ANALYSIS.

The following is a list of bodies which are taken into consideration here :—

Carbon,
Sulphur,
Alumina,
Barium Sulphate,
Strontium Sulphate,
Calcium Sulphate,
Calcium Fluoride,
Chromic Iron [$(FeMg)O \cdot (CrAl)_2O_3$],
Lead Chloride,
Lead Sulphate,
Silver Chloride,
Silica,
Silicates,
Stannic Oxide.

Besides these, there are bromide and iodide of silver, and certain aluminates, arseniates, fluorides, and metaphosphates, but they are of less frequent occurrence.

PRELIMINARY EXAMINATION.

This may be omitted if there is only a small quantity of the substance.

1. Heat a portion in an ignition tube. If **sulphur** sublimes, that element is present.

2. If the substance is black, this indicates generally the presence of **carbon** (coal, graphite, &c.). Heat a portion on platinum foil over the blowpipe flame; if the black matter burns away, it was carbonaceous. Graphite is recognised by its marking paper; it will not burn without the aid of oxygen.

3. Warm a portion with a strong solution of cyanide of potassium for a short time, filter, and test the filtrate with sulphide of ammonium. A precipitate indicates **silver.**

4. If a residue remains in 3, wash it completely. If it is white, moisten it with sulphide of ammonium; a blackening in-

dicates lead. If the residue is black already, heat it with acetate of ammonium and a little acetic acid, filter, and test the filtrate with hydrosulphuric acid for **lead.***

ACTUAL ANALYSIS.

If the absence of lead, silver, or sulphur has been demonstrated, of course the processes for separating them may be omitted.

To extract the lead, heat the substance repeatedly with fresh portions of a strong solution of acetate of ammonium, and filter.

f_1 Divide into three parts.

1. Acidify with nitric acid and add nitrate of silver. A precipitate indicates **chlorine.**

2. Acidify with hydrochloric acid and add chloride of barium. A precipitate indicates **sulphuric acid.**

3. Add sulphuric acid in excess. A precipitate indicates **lead.**

p_1 Wash it. To extract the silver, digest repeatedly with fresh portions of solution of cyanide of potassium at a gentle heat (if sulphur is present, in the cold), and filter.

f_2 Divide into two parts, the first large and the second small.

1. Add sulphide of ammonium and filter off the sulphide of **silver.** Test it by dissolving in nitric acid, diluting, and adding hydrochloric acid.

2. Acidify with hydrochloric acid and add chloride of barium. A precipitate indicates **sulphuric acid.**†

p_2 Wash and dry it. To drive off the **sulphur,** heat in a covered porcelain crucible. Then fuse with four parts of carbonate of potassium and sodium and one part of nitre in a platinum crucible, and pour out the fused mass. Powder it finely, boil it well with water, and filter.

f_3 Divide into four parts.

1. Test for **sulphuric acid.**

2. Test for **fluorine.**

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* The lead in glass will not be detected here.

† Derived from sulphates of barium, strontium, or calcium, which may have been more or less decomposed by the carbonate of potassium contained in the cyanide of potassium.

3. If yellow, **chromium** is present. Confirm by acidifying with acetic acid, and adding acetate of lead.

4. Add hydrochloric acid and evaporate to dryness, then take up again with hydrochloric acid and water. An insoluble *residue* indicates **silica.** Test the *solution* for **metals** whose oxides are soluble in alkalies.

p_3 Wash till the washings give no precipitate with chloride of barium. Dissolve in hydrochloric acid and test for **metals** in the usual manner.* If a residue still remains, it may be silica, sulphate of barium, fluoride of calcium, or (if dark-colored) chromic iron. As regards fluoride of calcium, it is readily decomposed by sulphuric acid. As regards chromic iron, the best way to decompose it is as follows:—Project the finely-powdered mineral into twelve times its bulk of fusing acid sulphate of potassium, stir often and fuse for half an hour, first gently, then at a strong heat, till fumes of sulphuric acid cease. Then add carbonate of sodium to about six times the amount of the mineral, fuse and add gradually nitrate of potassium in quantity equal to that of the carbonate of sodium; after some time increase the heat, stirring diligently with a platinum wire. When cool, boil with water.

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* If much silica has been found, first separate what may be present here by evaporating the solution to dryness and taking up again with hydrochloric acid and water, and then proceed to test for metals.

SPECIAL ANALYSIS.

SPECIAL ANALYSIS.

Analysis of Alloys.

Reduce to a fine state of division (by filing, boring, cutting, or pounding), and treat with a mixture of equal parts of strong nitric acid and water, till all action has ceased; add water, and filter.

The residue may contain stannic oxide, oxide of antimony, and a little arsenic oxide. Wash and treat it according to p. 74, 5.

The filtrate. Evaporate a drop or two to dryness, to discover if anything has been dissolved. If so, evaporate off the greater part of the excess of nitric acid, and examine for metals in the usual manner.

Note.—Tin is converted by nitric acid completely into insoluble stannic oxide. As regards the action of nitric acid on antimony, the dilute acid converts it almost entirely into antimonious oxide; the boiling strong acid converts it almost entirely into antimonic oxide, and generally the more concentrated the acid the more antimonic oxide is formed. Neither of the oxides of antimony is altogether insoluble in nitric acid; traces of antimony may therefore pass completely into solution. Arsenic oxide is soluble, but a portion is liable to combine with tin and antimony oxides.

Analysis of Silicates.

Silicious minerals may be divided for analytical purposes into two classes; first, silicates readily decomposable by acids (hydrochloric, nitric, sulphuric); second, silicates not readily decomposable by acids. Many rocks are mixtures of silicates belonging to both classes.

To ascertain to which of these two classes a given silicate belongs, reduce it to an impalpable powder, and digest a portion with strong hydrochloric acid at a temperature near the boiling point. If this fails to decompose it, try another portion by long-continued heating, with a mixture of three parts of strong sulphuric acid and one part of water. If this also fails, the silicate belongs to the second class. Whether decomposition has been effected or not may generally be seen at once, as in the former case a colored solution almost always forms, and the separated, gelatinous, flocculent, or finely pulverulent silica takes the place of the original heavy powder which grated under the glass rod with which it was stirred. Whether the decomposition is complete, or extends only to one of the components of a rock, may be ascertained by boiling the separated silica (after it has been washed) with a solution of carbonate of sodium. If a clear solution is obtained, complete decomposition has been effected; if not the decomposition is only partial.

In whichever way the substance is analysed, it must first be reduced to an impalpable powder.

SILICATES READILY DECOMPOSABLE BY ACIDS.

Silicates Decomposable by Hydrochloric Acid or by Nitric Acid.[*]

1. Digest the substance with hydrochloric acid at a temperature near the boiling point, until complete decomposition is effected, filter a small portion, and reserve the filtrate for subsequent examination; evaporate the rest of the fluid and precipitate to dryness, and heat the residue to a temperature barely exceeding 100°, breaking up the lumps with a pestle, until no more fumes of hydrochloric acid escape; allow to cool, moisten the residue with hydrochloric or nitric acid, as the case may be, add water, warm and filter. Examine the filtrate for **metals** in the usual way. Wash the residue; it consists of **silica,** but it can never be considered pure without further examination. It frequently contains **titanic acid,** and occasionally also barium and strontium sulphates and alumina. It is best tested by being heated in a platinum dish repeatedly with hydrofluoric and sulphuric acids till all the silica is volatilized. The residue is finally ignited, fused with acid sulphate of potassium, and treated with cold water. If a residue remains, it is examined for barium (and strontium).

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[*] Nitric is to be preferred where lead or silver is present.

The aqueous solution is diluted and tested for titanic acid* by long boiling; the filtrate from the latter is tested for aluminium by ammonia. (If it is possible that any chloride of silver has separated with the silica, digest a portion of the latter with ammonia, filter, and test the filtrate by supersaturating it with nitric acid.)

2. **Carbonates** are detected in the original treatment with hydrochloric acid. If **sulphides** are also not found in the same manner, they may be tested for with hydrochloric acid and zinc (p. 78, 4).

If the separated silica is black, and turns subsequently white on ignition, this indicates the presence of **carbon** or organic substances.

Examine the portion of the hydrochloric acid solution filtered off before evaporating, as follows:—Test one portion for **sulphuric acid.** Test another portion for **arsenic acid** by heating it to 70°, passing sulphuretted hydrogen into it, and examining the precipitate formed. Test a third portion for **phosphoric acid** by evaporating with addition of nitric acid to dryness on the water-bath, warming the residue with nitric acid, filtering, and adding molybdate of ammonium solution to the filtrate. When arsenic is found, use the filtrate from the sulphide of arsenic to test for phosphoric acid.

Boracic acid is best detected by fusing a portion of the substance in a platinum crucible lid with carbonate of potassium and sodium, boiling with water, and testing with turmeric paper.

In many cases boiling with water is sufficient to dissolve the **chlorides** present, but the safest way is to dissolve the silicate in nitric acid. The chlorides when dissolved are detected, of course, with nitrate of silver.

Fluorides are detected by the method given in p. 87, 3.

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* If the silica has been separated by evaporation on the water-bath, only a portion of the titanic acid is found with it; the other portion—often the larger—passes into the hydrochloric acid solution, and is precipitated with the iron and aluminium on the addition of ammonia. In order to find this latter portion, fuse the dried precipitate with acid sulphate of potassium, dissolve in cold water, filter if necessary, dilute largely, pass hydrosulphuric acid till all the iron is converted into the dyad state, and (without filtering off the sulphur) keep the fluid boiling for half an hour, passing carbonic acid through it all the while. Filter, wash, and ignite; the sulphur will burn off, the titanic acid will remain. Should it still contain iron, dissolve it again by fusing with acid sulphate of potassium and treating with cold water, and precipitate it by boiling with hyposulphite of sodium.

Silicates which Resist the Action of Hydrochloric Acid, but are Decomposed by Sulphuric Acid.

Heat the silicate with a mixture of three parts of strong sulphuric acid and one part of water (preferably in a platinum dish), finally drive off the greater portion of the sulphuric acid, boil the residue with hydrochloric acid, dilute, and filter. Examine the filtrate for **metals** in the usual way. The residue consists of the **silica,** and may also contain sulphate of barium, &c.; examine it by p. 128, 1. To test for **other bodies** treat a portion of the substance according to the method given below.

SILICATES NOT READILY DECOMPOSABLE BY ACIDS.*

1. Fuse the silicate with six parts of carbonate of potassium and sodium till no more gas is evolved. Pour it out and reserve a portion for the examination for acids. Put the rest into a porcelain dish, add water and hydrochloric acid, and heat gently till nothing but flocks of silica remain undissolved, evaporate to dryness, and treat the residue as directed p. 128, 1, for **silica** and all the **metals** except potassium and sodium.

2. Mix the silicate with five parts of pure, finely pulverized fluoride of calcium, stir the mixture in a platinum crucible with strong sulphuric acid to a thickish paste, and heat gently for some time under a good draught, finally increase the heat till the excess of sulphuric acid is driven off. Boil the residue with water, add chloride of barium as long as a precipitate continues to form, then baryta water to alkaline reaction, boil, filter, mix the filtrate with carbonate of ammonium and ammonia, filter, evaporate and ignite the filtrate, and examine for **potassium** and **sodium** in the usual way.

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* It will be understood, from what has been stated, that these are not decomposed by heating with hydrochloric acid and sulphuric acid in open vessels; but by heating them, reduced to a fine powder, in a sealed glass tube, with a mixture of three parts of concentrated sulphuric acid and one part of water, or with hydrochloric acid, to between 200° and 210°, most of them are decomposed, and may accordingly be analyzed also in this manner (AL. MITSCHERLICH).

3. Boil the portion of the fused mass reserved in 1, with water, and filter. Divide the filtrate into four parts, and test them as follows:—

1. Acidify with nitric acid and add nitrate of silver, for **chlorine.**
2. Test for **boracic acid** with turmeric paper.
3. Test for **fluorine** according to p. 88, 4.
4. Acidify with hydrochloric acid. Test a portion for **sulphuric acid** with chloride of barium. Heat the rest to 70°, and pass sulphuretted hydrogen to test for **arsenic acid.** If a precipitate is formed, filter it off, add nitric acid to the solution, and evaporate to dryness, treat the residue with nitric acid and water, and test the solution for **phosphoric acid** with molybdate of ammonium.

Analysis of Mineral Watres.*

The following bodies may be present:—

METALS.—Iron, Manganese, Thallium, Aluminium, Barium, Strontium, *Calcium*, *Magnesium*, Potassium, *Sodium*, Ammonium, Lithium, Cæsium, Rubidium.

ACIDS.—Hydrosulphuric, *Hydrochloric*, Hydrobromic, Hydriodic, Nitric, Nitrous, Boracic, *Carbonic*, Hydrofluoric, Phosphoric, *Silicic*, *Sulphuric*.

ORGANIC MATTER.

Of the above metals and acids, those in italics occur in most waters in considerable proportions: iron and hydrosulphuric acid occur sometimes in considerable proportions, the rest are only found in traces.

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* On this subject the following works by the author may be referred to:—

"Chemische Untersuchung der wichtigsten Mineralwasser des Herzogthums Nassau." Wiesbaden: Kreidel. 1850–1860. This work comprises seven analyses.

"Untersuchung der Mineralquellen zu Wildungen." Arolsen: Mittler. 1860.

"Analyse des Kaiser- und des Ludwigsbrunnens, wie der Elisabethenquellen zu Homburg v. d. H." Wiesbaden: Kreidel. 1863 and 1864.

"Analyse der Trinkquelle, der Badequelle und der Helenenquelle zu Pyrmont." Arolsen: Speyer. 1865.

"Analyse der Driburger und Herster Quelle." Wiesbaden: Kreidel. 1866.

There are also sometimes present oxygen, nitrogen, marsh gas, hyposulphurous acid, formic acid, propionic acid. But these are not considered here, as their detection usually comprises their quantitative estimation.

The analyst has moreover to examine the muddy ochreous deposits or solid sinter-deposits* of the spring, or possibly the residue left upon the evaporation of very large quantities of the water for lead, copper, antimony, arsenious and arsenic acids, nickel, cobalt, and other heavy metals. The greatest care is required in this examination to make sure that any heavy metals found do not proceed from pipes, stopcocks, &c. And of course the reagents used must be tested with equal care.

OPERATIONS AT THE SPRING.

1. Filter the water, if it is not perfectly clear, through Swedish paper, and collect the filtrate in large stoppered bottles. The residue on the filter is also reserved for examination. It may contain not only the matter suspended in the water, but also those constituents which separate at once upon coming in contact with the air (ferric hydrate, phosphate, silicate and arseniate).

2. The presence of **free carbonic acid** is usually sufficiently visible to the eye. However to convince yourself by positive reactions you may test the water with tincture of litmus and with lime water. If carbonic acid in the free state is present, the former acquires a wine-red color; the latter produces turbidity, which must disappear upon addition of the mineral water in excess.

3. **Free hydrosulphuric acid** is most readily detected by the smell. For this purpose half fill a bottle with the water, cover with the hand, shake, and then smell. In this way distinct traces of hydrosulphuric acid are often found which would escape detection by reagents. However, if you wish to have some visible reactions, fill a cylinder with the water, add a few drops of solution of acetate of lead in potash, place the cylinder on a sheet of white paper, and look down through the

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* These are the deposits which form in connection with most mineral springs, by the action of the air on the water.

water. Or half fill a large bottle with the water, and close with a cork to which is attached a slip of paper on which some acetate of lead and then carbonate of ammonium have been dropped; shake the bottle gently from time to time, and observe whether the paper is colored in the course of a few hours. Should the first experiment with acetate of lead indicate hydrosulphuric acid, while the second experiment with acetate of lead fails to indicate it, this shows that the water contains sulphide of potassium or sodium, but no free hydrosulphuric acid.

4. Mix a wineglassful of the water with tincture of galls. If an inky color is produced, dyad **iron** is present. The coloration makes its appearance only after some time, and increases in intensity from the top towards the bottom.

OPERATIONS IN THE LABORATORY.

EXAMINATION FOR THOSE CONSTITUENTS WHICH ARE PRESENT IN LARGER QUANTITIES.

Evaporate a litre or two of the water to dryness,* and heat the residue to faint redness. Blackening indicates **organic matter.** Mix the residue thoroughly, that it may have the same composition throughout. Boil a small portion with hydrochloric acid, filter, and test the filtrate for **sulphuric acid.** Boil a second small portion with nitric acid, filter, and test the filtrate for **hydrochloric acid.** Treat the rest of the residue with hydrochloric acid (an effervescence indicates **carbonic acid**), and boil repeatedly with fresh quantities of the acid till all that is soluble has dissolved; then filter. Wash the residue, and test it for **silica** in the microcosmic bead.

Heat the solution, and add to it a slight excess of ammonia. If a precipitate forms, filter it off; it may contain ferric hydrate, alumina, and phosphates, but need not be regarded here. Now add more ammonia, and oxalate of

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* The evaporation may be commenced directly over the flame, but should be finished on a water-bath. A large silver dish may be used, or in default of this a porcelain dish. If a porcelain dish is used, when the water has been reduced to a small bulk, it should be transferred with the deposit, if any, to a platinum dish; should the deposit adhere to the porcelain, it may be removed with a platinum knife.

ammonium in excess; filter off the precipitate of oxalate of **calcium.** Divide the filtrate into two parts. To the first add phosphate of sodium, stir well, and allow to stand twelve hours; a precipitate* indicates **magnesium.** Evaporate the second part to dryness, remove magnesium, if present, in the usual way, and test for **sodium** and **potassium.**

EXAMINATION FOR THOSE CONSTITUENTS WHICH ARE PRESENT IN MINUTE QUANTITIES.

1. To two or three gallons of the water add pure carbonate of potassium in slight excess, evaporate as directed p. 133, *foot-note,* and ignite the residue to faint redness. Mix the residue thoroughly, and then divide it into three parts, *a* and *b* each about a quarter, and *c* a half.

a. Warm with water, add nitric acid, digest for some time at a temperature near the boiling point, filter, and test for **phosphoric acid** with molybdate of ammonium.

b. Heat with water, add chloride of calcium as long as a precipitate continues to form, allow to settle, and collect the precipitate on a filter. Wash it, dry, ignite, transfer to a dish, add water, then excess of acetic acid, evaporate to dryness on the water-bath, heat till there is no more smell of acetic acid, add water, warm again, collect the insoluble residue on a filter, wash, dry, ignite it, and examine for **fluorine** by the etching test.

c. Boil repeatedly with water, filter, and wash the residue with boiling water.

The residue:

Consists chiefly of calcium and magnesium carbonates, silica, and—in the case of chalybeate waters—ferric oxide. It may also contain barium, strontium, aluminium, manganese, and titanic acid. Treat a portion with water in a dish, add hydrochloric acid to slightly acid reaction, then four or five drops of dilute sulphuric acid, evaporate to dryness, moisten with hydrochloric acid, add water, warm gently, filter, and wash the residue.

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* This precipitate is often visible only on the sides of the vessel when the fluid has been poured out.

p_1 consists principally of silica; it may also contain sulphate of barium, titanic acid, and carbon. Heat in a platinum dish repeatedly with hydrofluoric and sulphuric acids, till all the silica is driven off; finally evaporate to dryness. If a residue remains, fuse it with acid sulphate of potassium, treat with cold water, filter, and test the solution for **titanic acid** by long-continued boiling. If, on treating the fused mass with water, a residue remained insoluble, wash it, and incinerate the filter. Expose the ash on a platinum wire to the reducing flame for some time, moisten it with hydrochloric acid, and test for **barium** in the spectroscope. (Strontium will hardly be found here.)

f_1. Transfer to a flask, add chloride of ammonium, ammonia till just alkaline, then sulphide of ammonium; fill the flask up to the neck, close it, and allow to stand twenty-four hours in a rather warm place. Filter.

p_2. Dissolve in nitric acid, boil, add pure soda in excess, boil again, and filter.

p_3. Test a part for **manganese** in the carbonate of sodium bead, and another part for **iron,** by dissolving in hydrochloric acid, and adding sulphocyanide of potassium.

f_3. Add excess of chloride of ammonium, and heat. A precipitate indicates **aluminium.***

f_2 contains generally the whole of the **strontium,** and possibly traces of barium. Precipitate it with carbonate of ammonium, filter after some time, wash the precipitate, dry and test it as directed p. 76, 3rd par.

The solution:

Evaporate to a very small bulk, allow to cool; place the dish in a slanting position that the small quantity of liquid may separate from the saline deposit; transfer a few drops of the fluid to a watch-glass, acidify very slightly with hydrochloric acid, and test with turmeric paper for **boracic acid.** Evaporate the contents of the dish to dryness, rubbing with a pestle, so that no moisture may remain. Divide the residual powder into two parts, one larger than the other.

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* It is no use testing for aluminium if a porcelain dish has been used in the evaporation.

Put the larger portion of the powder into a flask, add alcohol of 90 per cent., boil in a water-bath and filter hot; repeat the operation with fresh alcohol twice. Mix the solution with a few drops of potash, and evaporate to dryness. Treat the residue three times with warm alcohol, filter, evaporate the filtrate to dryness with addition of a drop of potash, dissolve in a little water, acidify with hydrochloric acid, add bisulphide of carbon, then chloride of soda, and shake; if **iodine** is present, add more chloride of soda cautiously till the **bromine,** if present, shows itself.

Warm the smaller portion of the powder with water, add hydrochloric acid to distinct acid reaction, evaporate *nearly* to dryness, then mix with alcohol of 90 per cent., and filter. Evaporate the filtrate to dryness and test for **lithium** in the spectroscope.

2. To some of the clear water add mercuric chloride and carbonate of potassium to test for **ammonium.** Or put a litre of the water into a retort, distil over 100 c. c. through a Liebig's condenser, and apply Nessler's test.

3. Evaporate some of the water to dryness, finishing on the water-bath, and test for **nitric acid** with brucia or sulphophenic acid.

4. To some of the water add a solution of iodide of potassium and starch,* and then some pure dilute sulphuric acid, and allow to stand a few minutes. A blue color indicates **nitrous acid.** It is well to try a counter experiment with pure water.

5. To twenty or thirty gallons of the water add carbonate of sodium till the reaction is just alkaline, and evaporate in a large iron vessel until the salts soluble in water begin to separate, then filter. The precipitate may be washed (the washings not being mixed with the filtrate) and examined by the method given below for sinter-deposits. Mix the filtrate with hydrochloric acid to acid reaction, heat, just precipitate the sulphuric acid with chloride of barium, filter off the precipi-

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* 1 part of iodide of potassium, 20 parts of starch, 500 parts of water.

tate, evaporate to dryness, digest with alcohol of 90 per cent., and examine for **cæsium** and **rubidium** according to the directions given under the head of the Detection of Rare Metals.

Dissolve the residue insoluble in alcohol in a little hot water, and add ammonia in excess; if a precipitate appears, filter it off. Add iodide of potassium to the warm solution, and allow to stand. If a precipitate forms, test it in the spectroscope for **thallium.**

EXAMINATION OF THE SINTER-DEPOSIT.

1. Remove sand or stones by sifting or elutriation, and soluble salts by washing. Then digest about 200 grm. with water and hydrochloric acid (effervescence indicates **carbonic acid**) at a gentle heat, until the soluble part is dissolved. Dilute, allow to cool, filter, and wash the residue.

The filtrate:

Divide into two portions, one larger than the other.

Heat the *larger portion* to 70°, and conduct hydrosulphuric acid into it for some time. Then allow to stand in a moderately warm place till the smell of hydrosulphuric acid has passed off, and filter.

> p_1. Wash and dry, remove the greater part of the free sulphur by bisulphide of carbon, warm gently with yellow sulphide of potassium, dilute, filter, and wash the residue with water containing a little sulphide of potassium.
>
> > p_2. Remove from the filter, boil with dilute nitric acid, filter, and wash the residue.
> >
> > > p_3. Treat with sulphuretted hydrogen water, so that sulphate of lead may not be overlooked, then test for **barium** and **strontium** by incinerating, holding the ash on a platinum wire in the reducing flame for some time, moistening with hydrochloric acid, and finally holding in the spectroscope.
> > >
> > > f_3. Add pure sulphuric acid, evaporate to dryness on the water-bath, treat with water, and filter. The *residue* consists of sulphate of **lead;** to make sure, wash it, and treat with sulphuretted hydrogen

water. To the *filtrate* add ammonia, to test for **copper.**

f_2. Precipitate with hydrochloric acid. Wash and dry the precipitate, extract with bisulphide of carbon, treat the filter and precipitate with red fuming nitric acid in a dish, evaporate off the greater part of the acid, add excess of carbonate of sodium and some nitrate of sodium, fuse and treat the fusion as directed p. 72, 1, for the detection of **tin, antimony,** and **arsenic.**

f_1. Evaporate a portion to dryness, treat with nitric acid and water, filter, and test the filtrate for **phosphoric acid** with molybdate of ammonium. Mix the remainder of the filtrate in a flask with chloride of ammonium, ammonia, and sulphide of ammonium, fill up to the neck, close, and allow to stand in a rather warm place until the supernatant fluid is yellow. Filter.

p_4. Wash with water containing sulphide of ammonium, treat with dilute hydrochloric acid, and examine for **nickel, cobalt, iron, manganese, zinc, aluminium** and **silica,** in the usual manner. Precipitate a portion of the hydrochloric acid solution with ammonia, and treat the precipitate as directed p. 128, 1, to examine for **titanic acid.**

f_4. Add carbonate of ammonium and oxalate of ammonium to precipitate **calcium, barium,** and **strontium,** and filter. Test the *precipitate* for barium and strontium, as directed p. 76, 3rd par. Test the *filtrate* for **magnesium.**

Dilute the *small portion* of the hydrochloric acid solution considerably, add chloride of barium, and allow to stand twelve hours, to test for **sulphuric acid.**

The residue:

This consists usually of sand, hydrated silica, clay, organic matter, and possibly also barium and strontium sulphates. Boil with carbonate of sodium to dissolve the hydrated **silica,** then treat on the filter with dilute hydrochloric acid to dissolve the barium and strontium, leaving the clay behind. Evaporate the solution to dryness, and test for **barium** and **strontium** in the spectroscope.

2. Ignite a separate portion of the deposit, stir in water, add acetic acid to acid reaction, evaporate until the acid is completely driven off, add water, heat, filter, wash, dry and ignite the residue, and examine it for **fluorine** by the etching test.

Analysis of the Ashes of Plants, Manures, &c.

The substance to be incinerated must first be carefully cleaned. The incineration is effected best in a muffle, but it may be conducted also in a crucible placed in a slanting position, or in a platinum dish, with the aid of a lamp chimney to create a draught. The heat must always be moderate, that the volatilization of certain constituents, especially chlorides, may be avoided. It is not always necessary to continue the combustion until the carbon is entirely consumed. With ashes containing a large proportion of fusible salts, it is advisable first to carbonize thoroughly, to extract the mass with water, and then to complete the incineration.

It is well to examine separately the aqueous extract, the hydrochloric acid extract of the residue, and the insoluble residue.

EXTRACTION WITH WATER, AND EXAMINATION OF THE SOLUTION.

Boil the ash with water, filter, and wash the residue. Divide the solution into six parts.

1. Heat, add hydrochloric acid in excess, and heat again. Effervescence indicates **carbonate** of potassium or sodium; smell of sulphuretted hydrogen indicates sulphide of potassium or sodium produced from a sulphate by the action of carbon; separation of sulphur and smell of sulphurous acid indicates a hyposulphite (occasionally found in the ash of coal). Filter if necessary, and test the filtrate for **sulphuric acid**.

2. Evaporate to a small bulk, add hydrochloric acid to acid reaction. Test a few drops with turmeric paper for **boracic acid.** Evaporate the rest to dryness, treat the residue with nitric acid and water, and filter. The residue consists of **silica.** To the filtrate add molybdate of ammonium to test for **phosphoric acid.**

3. Add nitrate of silver, as long as a precipitate forms,

warm, and add ammonia; if a black residue remains, this consists of sulphide of silver, and is to be filtered off. Mix with nitric acid in slight excess, and collect the precipitate on a filter. It contains the **chlorine** (bromine and iodine). If you want to test it for bromine and iodine treat it with zinc and sulphuric acid, filter, add bisulphide of carbon and a drop or two of chloride of soda, and shake; if iodine is present, add more chloride of soda cautiously, and shake, when the iodine color will disappear, and the bromine color will take its place.*

4. Heat with hydrochloric acid, add excess of ammonia, and oxalate of ammonium, and allow to stand. A precipitate indicates **calcium.** Filter it off, and test for **magnesium** by adding phosphate of sodium, and allowing to stand twelve hours. Traces of magnesium are often found; calcium is only met with in exceedingly minute quantities.

5. Examine for **potassium** and **sodium** in the usual way.

6. Test for **lithium** and **rubidium.** See Detection of Lithium, &c., in Presence of Potassium and Sodium.

EXTRACTION WITH HYDROCHLORIC ACID, AND EXAMINATION OF THE SOLUTION.

If the residue insoluble in water contains much carbon, incinerate it further. Test a small portion for **fluorine** (p. 87, 3). Warm the rest with hydrochloric acid. Effervescence indicates **carbonate** of calcium or magnesium; smell of chlorine indicates manganese. Evaporate to dryness with a little sulphuric acid, and heat a little more strongly, then moisten with hydrochloric acid and nitric acid, add water, warm and filter. Test the residue for **barium** and **strontium** (p. 135, p_1). Divide the filtrate into two parts.

1. Add hydrosulphuric acid. If the precipitate is other than quite white you must examine it in the usual way.

2. Mix with carbonate of sodium till a precipitate just begins to be formed, then add acetate of sodium and some acetic acid, and filter.

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* Iodine in plants is best detected by dipping the plant in weak potash, drying, incinerating, treating with water, evaporating the solution to absolute dryness, exhausting with alcohol, and proceeding as directed p. 136, top.

p_1. A white precipitate of **ferric phosphate** usually forms, which may contain also phosphate of aluminium. Wash, heat with pure soda, filter, acidify filtrate with hydrochloric acid, add slight excess of ammonia, and warm; a precipitate indicates **aluminium.**

f_1. If not reddish, add ferric chloride drop by drop till a red color is produced. Heat to boiling, filter hot,* add ammonia to the filtrate, then sulphide of ammonium; allow to stand in a flask filled to the mouth and closed, and filter after some time.

p_2. Examine for **manganese** and **zinc.**†

f_2. Examine for **calcium** and **magnesium** in the usual way. The calcium precipitate may contain strontium, which may be tested for as directed p. 76, 3rd par.

EXAMINATION OF THE RESIDUE EXHAUSTED BY WATER AND HYDROCHLORIC ACID.

Most ashes will entirely dissolve in hydrochloric acid with the exception of a little silica, which may separate, and of extraneous matter such as sand, clay, and carbon. In most cases, therefore, you have only to boil the residue with carbonate of sodium, filter hot, wash with boiling water, and test for **silica** in the filtrate by evaporation with hydrochloric acid.

Some ashes, however, contain large quantities of silica, and silicates might be present in them which are not decomposed by hydrochloric acid. If you want to examine the residue you may divide it in half. Evaporate *one half* to dryness with pure soda in a silver or platinum dish. This decomposes the silicates, leaving the sand. Acidify now with hydrochloric acid, evaporate to dryness, &c., and proceed as above under the "Examination of the Hydrochloric Acid Extract." For the detection of alkalies use the *other half* of the residue, treating it as directed p. 130, 2.

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* If the filtrate is colored, it must be boiled with more acetate of sodium.

† This metal is occasionally found.

Analysis of Compound Cyanides* insoluble in Water, or of Mixtures containing them.

The presence of cyanogen in such substances is usually indicated by the smell of hydrocyanic acid on heating with hydrochloric acid.

Were you to attempt to dissolve them in acids, and analyse them in the usual way, you would meet with confusing results.

If you simply wish to examine for metals, treat the powdered substance with strong sulphuric acid diluted with a little water, and ignite till the excess of acid is driven off. The residue consists of sulphates, and may be analysed in the usual manner.

But if you wish to analyse the substance completely, the following method must be adopted.

1. Extract the substance with water, boil the residue with potash for a few minutes, then add carbonate of sodium and boil again for some time. Filter and wash the residue.

p_1. Will be free from cyanogen unless cyanide of silver is present. Examine in the usual way for mixed substances.

f_1. Add a sufficient quantity of hydrosulphuric acid to test for copper, iron, &c.†

If no permanent precipitate is formed, lead and zinc are absent, pass on to f_2.

If a permanent precipitate is formed, add a little yellow sulphide of sodium drop by drop till the copper, iron, &c., are just precipitated, heat gently, filter, and wash the precipitate.

p_2. Dissolve in nitric acid (**mercuric** sulphide may be left), and examine for **lead, copper, zinc,** and other metals of the third group which may have dissolved by the agency of organic matter.

f_2. Add water, then sulphuric acid in excess, and

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* The analysis of simple cyanides is straightforward enough, as they are all decomposed by boiling with hydrochloric acid, hydrocyanic acid being evolved, and the metal converted into chloride.

† Do not add the hydrosulphuric acid until the liquid smells of it, as this might lead to the precipitation of aluminium, and even of tin, antimony, and arsenic, which are not intended to be thrown down here.

hydrosulphuric acid if the fluid already does not smell strongly of it. Filter.

p_3. Wash, test for **mercury, tin, antimony,** and **arsenic** in the usual way.

f_3. May contain aluminium, cyanogen acids (containing cobalt, manganese, iron, chromium), and other acids. Divide into two parts.

1. Test for **acids.**

2. Evaporate nearly to dryness, add strong sulphuric acid, and heat, till the greater portion of the excess of acid is driven off. Dissolve the residue in water, and test the solution for **cobalt, manganese, iron, chromium,** and **aluminium,** in the usual way.

2. Decompose another portion of the original substance by ignition with strong sulphuric acid, and test the residue for **potassium** and **sodium,** after having separated the other metals.

General Rules for the Detection of Inorganic Substances in the presence of Organic Matter.

a. The substance dissolves in water, but the solution is dark-colored or slimy.

1. Boil a portion of the solution for some time with nitric acid, and filter. If the organic matter is destroyed, use the solution for analysis in the ordinary way. If the organic matter is not destroyed, test the solution only for silver, potassium, and chlorine.

2. If the organic matter has not been destroyed in 1, heat a fresh portion of the original solution with hydrochloric acid on a water-bath, gradually add chlorate of potassium until the mixture is decolorized and perfectly fluid. Heat until it no longer smells of chlorine, then dilute and filter. Examine the filtrate in the usual way.

3. Aluminium and chromium, being not precipitated in the ordinary course of analysis when non-volatile organic matter is present, must be looked for separately. Mix a fresh portion of the original solution with carbonate of sodium and chlorate of potassium, and throw the mixture gradually into a red-hot crucible. Treat the fused mass with water, and examine the

solution for chromic acid and aluminium, the residue for aluminium.

4. Test a fresh portion of the original substance for ammonium.

b. The substance does not dissolve completely when treated with boiling water, but the liquid can be filtered.

Filter, examine *the filtrate* in the usual way; unless it requires decoloration, when the process directed in *a* must be adopted. If *the residue*

is fatty. Remove the fatty matter with ether. If a residue still remains, examine by the usual method for mixed substances.

is resinous. Remove the resin with alcohol, or extract with alcohol first and then with ether.

is neither fatty nor resinous (such as woody fibre). Dry and divide it into three parts.

1. Incinerate* and examine the ash by the ordinary method for mixed substances.
2. Examine for metals which may be volatilized by ignition, such as mercury, cadmium, arsenic, zinc, and for acids as directed in *a*.
3. Test for ammonium.

c. The substance does not completely dissolve when treated with boiling water, and the liquid cannot be filtered.

Treat the substance like the residue in *b*.

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* It is often advisable first to carbonize thoroughly, then to extract with water and incinerate the residue, the aqueous extract and ash being both examined.

REACTIONS AND SEPARATION
OF
RARE INORGANIC BODIES.

THE RARE METALS.

The rare metals may be classed as follows, see p. 25 :—

PRECIPITATED WITH METALS OF GROUP I.

As acids	Niobium. Tungsten.
As chloride	Thallium.

PRECIPITATED WITH METALS OF GROUP II. AS SULPHIDES.

Div. I.—Sulphides insoluble in sulphide of ammonium.

Palladium.
Rhodium.
Osmium.
Ruthenium.

Div. II.—Sulphides soluble in sulphide of ammonium.

Gold.
Platinum.
Iridium.
Molybdenum.
Tellurium.
Selenium.

PRECIPITATED WITH METALS OF GROUP III.

As sulphides	(Thallium). Uranium. Indium.

As hydrates	Beryllium. Thorium. Zirconium. Yttrium. Terbium. Erbium. Cerium. Lanthanium. Didymium. Titanium. Tantalum.

PRECIPITATED BY HYDROCHLORIC ACID IN THE FILTRATE FROM METALS OF GROUP III., AS SULPHIDES.

Vanadium.
(Tungsten).

FOUND AMONG METALS OF GROUP V.

Lithium.
Cæsium.
Rubidium.

REACTIONS OF RARE METALS.

Niobium.

Niobium forms two oxides, the niobous and niobic anhydrides. The former is found in columbite, and samarskite. Niobous anhydride is white; it varies in density between 4·6 and 6·5; it combines with bases and acids. Strong sulphuric acid dissolves niobous anhydride upon heating; by addition of a large quantity of cold water, a clear solution is obtained, from which the niobous anhydride separates in combination with sulphuric acid, gradually in the cold, rapidly on boiling; upon washing the precipitate with carbonate of ammonium, and then with highly dilute hydrochloric acid, the niobous acid is obtained; and upon igniting the precipitate in an atmosphere of carbonate of ammonium, niobous anhydride is obtained. By fusion with acid sulphate of potassium, niobous anhydride is readily dissolved; on treating the fused mass with water, sulphate of niobous anhydride is left behind. With potash, niobous anhydride fuses to a clear mass, soluble in water. With soda, niobous anhydride fuses to a turbid mass, from which water dissolves the excess of soda; after the removal of the latter, the niobite of sodium dissolves in water. Fusion with carbonate of sodium has the same effect as fusion with soda. By ignition of niobous anhydride, mixed with charcoal, in a current of chlorine, white niobous chloride and yellow niobic chloride are formed; both these chlorides are solid, but the latter is somewhat more volatile than the former. Microcosmic salt dissolves niobous anhydride copiously; the bead produced in the inner flame shows a violet, blue, or brown color; addition of ferrous sulphate imparts a blood-red tint to it. The solutions of the alkaline niobites show the following reactions:—

They are not rendered turbid by *boiling.*

Hydrochloric acid produces a precipitate which does not redissolve in excess of the reagent (here niobous acid differs from titanic and tantalic acids).

Ammonia and *sulphide of ammonium* added to acidified solutions produce a precipitate of acid niobite of ammonium.

Soda slowly added to solution of niobite of sodium precipitates the salt in the crystalline form.

Sulphuric acid precipitates the whole of the niobous acid on boiling.

Carbonic acid throws down acid niobite of the alkali metal, which is soluble in boiling dilute solution of carbonate of sodium. (Means of separating niobous acid from tantalic acid.)

Ferrocyanide of potassium produces a dark brown precipitate in acidified solutions.

If hydrochloric acid is added and then *zinc,* the precipitate of niobous acid acquires a blue tint, which gradually changes to brown (here niobous acid differs from tantalic acid).

Tungsten.

Tungsten occurs most frequently as tungstate of calcium and the mineral wolfram. Tungsten, obtained by the reduction of tungstic anhydride in a stream of hydrogen at an intense red heat, is an iron-grey powder, which fuses with extreme difficulty. The pulverulent metal is converted by ignition in the air into tungstic anhydride (WO_3), by ignition in a current of dry air-free chlorine into dark violet hexachloride, which sublimes, and a more volatile red compound ($WCl_4 . WCl_6$). These chlorides are decomposed by water into the corresponding hydrates of tungsten with formation of hydrochloric acid. Tungsten is insoluble in acids, even in aqua regia, and also in potash; it dissolves, however, in the latter if mixed with chloride of soda. Binoxide of tungsten is black; by intense ignition with free access of air it is converted into tungstic anhydride. Tungstic anhydride is lemon-yellow, fixed, insoluble in water and acids. By fusing tungstic anhydride with acid sulphate of potassium, and treating the fused mass with water, an acid solution is obtained, which contains no tungstic acid; after the removal of this solution the residue, consisting of tungstate of potassium and excess of tungstic acid, completely dissolves in water containing carbonate of ammonium (means of separating tungstic from silicic acid). Tungstates of the alkali metals soluble in water are formed readily by fusion with alkaline carbonates, but with difficulty by boiling with solutions of the same. In analysing wolfram, it is fused with alkaline carbonates, and treated with water, which dissolves the alkaline tungstate formed. Microcosmic salt dissolves tungstic anhydride; the bead acquiring in the reducing flame a blue color, which turns blood-red on the addition of ferrous sulphate. The solutions of tungstates show the following reactions:—

Hydrochloric acid, nitric acid, and sulphuric acid, produce a white precipitate, which is insoluble in excess of the acids (here tungstic acid differs from molybdic acid), but soluble in ammonia. Upon evaporating with excess of hydrochloric acid to dryness, and treating the residue with water, tungstic anhydride is left undissolved.

Hydrosulphuric acid barely precipitates acid solutions.

Sulphide of ammonium fails to precipitate solutions of alkaline tungstates; upon acidifying the mixture, light-brown tersulphide of tungsten precipitates, which is slightly soluble in pure water, but insoluble in water containing salts.

Ferrocyanide of potassium, added to acidified solutions, colors the fluid deep brownish-red, and after some time produces a precipitate of the same color.

Stannous chloride produces a yellow precipitate; on acidifying with hydrochloric acid, and applying heat, this precipitate acquires a beautiful blue color. (Highly characteristic and delicate.)

Zinc added to solutions of alkaline tungstate, mixed with phosphoric acid, imparts a beautiful blue color.

Chloride of barium, and *nitrate of silver* produce white precipitates.

Thallium.

This metal occurs in small quantities often in copper pyrites, iron pyrites, and crude sulphur. It is also found accumulated in the dust of the flues leading to sulphuric acid chambers, where the furnaces are fed with thalliferous pyrites. It is sometimes found in sulphuric acid and hydrochloric acid, lepidolite, preparations of bismuth and cadmium, ores of zinc mercury and antimony, the ashes of plants, and certain saline waters. There are two oxides—thallous oxide (Tl_2O, the base of the salts) and sesquioxide of thallium. Thallous oxide is colorless and fusible; when fusing it acts on glass or porcelain. It is soluble in water; the solution is colorless, alkaline, caustic, and absorbs carbonic acid. It is also soluble in alcohol. Sesquioxide of thallium is insoluble in water and dark violet; its hydrate is brown. Sesquioxide of thallium is hardly acted on by concentrated sulphuric acid in the cold; on heating it is dissolved. On heating it with hydrochloric acid, thallous chloride is formed with evolution of chlorine. The thallium salts are colorless, some are soluble in water (sulphate, nitrate, phosphate, tartrate, acetate), some are difficultly soluble (carbonate, chloride), some nearly insoluble (iodide, &c.). Thallium compounds color flame intensely green; the spectrum shows only one characteristic splendid emerald green line. If the quantity of metal is small, the line soon disappears. The spectroscope generally affords the best means of detecting thallium. Thalliferous pyrites often gives the green line at once. If you want to look for thallium in crude sulphur, it is best to remove the greater part of the sulphur with bisulphide of carbon, and then to test the residue. In the presence of much sodium with very small quantities of thallium the green line will not be seen, unless you moisten the substance and examine the spectrum which is first produced. Thallium salts give the following reactions:—

Hydrochloric acid precipitates thallous chloride, if the solution is not very dilute; the precipitate is white and readily subsides; light has no action upon it; it is less soluble in dilute hydrochloric acid than in water.

Hydrosulphuric acid does not precipitate acid solutions.

Sulphide of ammonium produces in neutral solutions a black precipitate of sulphide of thallium, which readily unites to lumps, especially on heating. The sulphide is insoluble in ammonia, sulphides of the alkali metals and cyanide of potassium, it dissolves in dilute hydrochloric and sulphuric acids with some difficulty, and it is readily soluble in nitric acid. On being heated it first fuses and then volatilizes.

Potash and ammonia produce no precipitate.

Alkaline carbonates precipitate the carbonate only from very concentrated solutions (for 100 parts of water dissolve 5·23 parts of the carbonate of thallium at 18°).

Iodide of potassium precipitates iodide of thallium, even from the most dilute solutions. The precipitate is light yellow, almost insoluble in water, not quite so insoluble in iodide of potassium.

Chloride of platinum precipitates chloride of thallium and platinum (Tl_2PtCl_6) from solutions which are not too dilute; the precipitate is of a pale orange color, and very difficultly soluble.

Zinc precipitates the metal in black laminæ.

For the detection of thallium in the wet way, iodide of potassium is the most delicate reagent; if iron is present, it must previously be reduced by sulphite of sodium.

Palladium.

Palladium is found in the metallic state, occasionally alloyed with gold and silver, but more particularly in platinum ores. It greatly resembles platinum, but is somewhat darker in color. It fuses with great difficulty. It does not oxidize when heated in the air. It is sparingly soluble in pure nitric acid, but dissolves somewhat more readily in nitric acid containing nitrous acid; it dissolves very sparingly in boiling concentrated sulphuric acid, but readily in aqua regia. It dissolves in fusing bisulphate of potassium. There are three oxides: the suboxide, the protoxide (the base of the ordinary salts), and the binoxide. Protoxide of palladium is black, the hydrate dark-brown; both are, by intense ignition, resolved into oxygen and metallic palladium. Binoxide of palladium is black; by heating with dilute hydrochloric acid, it is dissolved to the ordinary chloride, with evolution of chlorine. The salts of palladium are mostly soluble in water; they are brown or reddish-brown; they are decomposed by ignition, leaving metallic palladium behind. Water precipitates from a solution of the nitrate containing a slight excess of acid a brown basic salt. Chloride of palladium shows the following reactions:—

Hydrosulphuric acid throws down black sulphide, insoluble in sulphide of ammonium, soluble in boiling hydrochloric acid, readily soluble in aqua regia.

Sulphide of ammonium produces the same precipitate.

Potash precipitates a brown basic salt, soluble in excess of the precipitant.

Ammonia produces a flesh-colored precipitate of $N_2H_6PdCl_2$, which is soluble in excess of the precipitant to a colorless fluid; on adding hydrochloric acid to this solution, a yellow crystalline body is thrown down, having the same composition as the first precipitate.

Cyanide of mercury precipitates yellowish-white gelatinous cyanide of palladium, soluble in hydrochloric acid and in ammonia. (Very characteristic.)

Stannous chloride produces, in the absence of hydrochloric acid, a brownish black precipitate; in the presence of hydrochloric acid, a red color, which speedily turns brown, and ultimately green, and upon addition of water brownish-red.

Ferrous sulphate produces a deposit of palladium on the sides of the glass.

Iodide of potassium precipitates black iodide of palladium. (Very characteristic.)

Chloride of potassium precipitates from strong solutions chloride of potassium and palladium (K_2PdCl_4), in the form of golden-yellow needles, which dissolve readily in water to a dark red fluid, but are insoluble in alcohol.

Nitrite of potassium produces a yellowish precipitate, which becomes reddish by long standing, and dissolves in a considerable quantity of water.

Rhodium.

Rhodium is found in small quantities in platinum ores. It is almost silver white, very malleable, and difficultly fusible. When prepared in the wet way it is a grey powder. The powder when ignited in the air absorbs oxygen, which it gives up again upon stronger ignition. Rhodium is insoluble in all acids; it dissolves in aqua regia only when alloyed with platinum, copper, &c., and not when alloyed with gold or silver. Fusing metaphosphoric acid and fusing bisulphate of potassium dissolve it, forming a rhodium salt. There are four oxides: the protoxide, sesquioxide (base of the salts), binoxide, and tetroxide (anhydride of a weak acid). The sesquioxide is grey; it is insoluble in acids, but dissolves in fusing metaphosphoric acid and in fusing bisulphate of potassium. There are two hydrates: a yellow and a brownish-black. The solutions are rose-colored.

Sulphuretted hydrogen precipitates in time, especially when assisted by heat, a brown sulphide, which is insoluble in sulphide of ammonium, but dissolves in boiling nitric acid.

Sulphide of ammonium produces the same precipitate under the same circumstances.

Potash, if added in not too large excess, throws down at once yellow RoH_3O_3,H_2O, which is soluble in excess of the precipitant at the ordinary temperature; on boiling the solution, blackish-brown RoH_3O_3 is precipitated. In a solution of the chloride, potash at first produces no precipitate, but, on addition of alcohol, black RoH_3O_3 separates.

Ammonia produces after some time a yellow precipitate, soluble in hydrochloric acid.

Zinc precipitates black metallic rhodium.

On heating with *nitrite of potassium,* the chloride of rhodium becomes yellow, and an orange-yellow precipitate is formed, which is slightly soluble in water, but readily soluble in hydrochloric acid; at the same time another portion of the rhodium is converted into a yellow salt, which remains in solution and is precipitated by alcohol.

All solid compounds of rhodium, on ignition in hydrogen, yield the metal, which is well characterized by its insolubility in aqua regia, its solubility in fusing bisulphate of potassium, and the behaviour of its solution to potash and alcohol.

Osmium.

Osmium is found in platinum ores as a native alloy of osmium and iridium. It is generally obtained as a black powder, or grey and with metallic lustre; it is infusible. The metal, the protoxide and the binoxide, oxidize readily when ignited in the air, and give osmic anhydride (OsO_4), which volatilizes, and makes its presence speedily known by its peculiar, exceedingly irritating and offensive smell, resembling that of chlorine and iodine (highly characteristic). If a little osmium on a strip of platinum foil is held in the outer mantle of a flame, the flame becomes most strikingly luminous. Even minute traces of osmium may by this reaction be detected in alloys of iridium and osmium; but the reaction is in that case only momentary; it may, however, be reproduced by holding the substance first in the reducing flame, then again in the outer mantle. Nitric acid, more particularly red fuming nitric acid, and aqua regia, dissolve osmium to osmic acid. Application of heat promotes the solution, which is however attended in that case with volatilization of osmic acid. Very intensely ignited osmium is insoluble in acids; it is fused with nitrate of potassium, and the fused mass distilled with nitric acid; the osmic acid is found in the distillate.

By heating osmium in dry chlorine free from air, first bluish-black bichloride is formed, but always only in small quantity, then the more volatile and red tetrachloride; if moist chlorine is used, a green mixture of both chlorides is formed. The bichloride dissolves with a blue color, the tetrachloride with a yellow color, and both together with a green color, which turns red. The solutions are soon decomposed, osmic acid, hydrochloric acid, and a mixture of protoxide and binoxide, being formed, the mixed oxides separating as a black powder. On heating a mixture of powder of osmium, or of sulphide of osmium, and chloride of potassium, in chlorine, a compound of tetrachloride of osmium and chloride of potassium is produced in the form of octahedra, which are slightly soluble in water and insoluble in alcohol. The solution of this double salt is more permanent than that of the tetrachloride. Potash decolorizes the solution; on boiling bluish-black, hydrated binoxide of osmium separates. On fusing the double chloride with carbonate of sodium, dark grey binoxide of osmium separates. The pure osmic anhydride has no effect on litmus, and cannot form salts. The pure anhydride is white, crystalline, fusible at a gentle heat, and boils at about 100°; the fumes attack the nose and eyes powerfully. Heated with water, it fuses and dissolves, but slowly. The solution has an irritating, unpleasant smell. Alkalies color the solution yellow in consequence of the formation of osmite of potassium (K_2OsO_4); on distilling, the greater part of the osmic acid passes over (very characteristic), the remainder splits into oxygen and osmious acid, or on boiling into osmic acid, binoxide of osmium, and potash. Osmic acid shows the following reactions:—

Hydrosulphuric acid precipitates brownish-black sulphide, which only separates when a strong acid is present; the precipitate is insoluble in sulphide of ammonium.

Indigo solution is decolorized.

Iodide of potassium is decomposed, iodine being separated.

Nitrite of potassium reduces osmate of potassium readily to osmite.

Sulphite of sodium produces a deep violet coloration, and dark-blue sulphite of osmium (corresponding to the protoxide) gradually separates, especially on evaporating or warming with sulphate or carbonate of sodium.

Ferrous sulphate produces a black precipitate of binoxide.

Stannous chloride produces a brown precipitate soluble in hydrochloric acid.

Zinc and many metals in the presence of a strong acid precipitate metallic osmium.

All the compounds of osmium yield the metal on ignition in a current of *hydrogen*.

Ruthenium.

Ruthenium is found in small quantities in platinum ores. It is greyish-white, brittle, and very difficultly fusible. It is barely acted on by aqua regia; fusing bisulphate of potassium fails altogether to affect it. By ignition in the air it is converted into dark bluish sesquioxide, insoluble in acids; by ignition with chloride of potassium in a current of chlorine, into terchloride of ruthenium and chloride of potassium; by fusion with nitrate of potassium, with potash or with chlorate of potassium, into ruthenate of potassium (K_2RuO_4). The fused mass obtained in the latter case is dark grey, and dissolves to an orange-colored fluid, which colors the skin black, from the reduction and separation of black oxide. Acids throw down from the solution black sesquioxide, which dissolves in hydrochloric acid to an orange-yellow fluid. The solution is resolved by heat into hydrochloric acid and dark brown sesquioxide, which remains suspended for a long time, and has great tinctorial power. In a concentrated state it gives with chloride of potassium and chloride of ammonium a crystalline, glossy-violet precipitate, which on boiling with water deposits black oxybichloride.

Hydrosulphuric acid gas causes at first no alteration, but after some time the fluid acquires an azure tint, and deposits brown sulphide of ruthenium (very characteristic).

Sulphide of ammonium produces a dark brown precipitate, barely soluble in excess of the precipitant.

Potash precipitates black hydrated sesquioxide, which is insoluble in alkalies, but dissolves in acids.

Sulphocyanide of potassium produces—in the absence of other metals of the platinum ores—after some time a red coloration, which gradually changes to purple, and, upon heating, to a fine violet tint (very characteristic).

Zinc produces at first an azure coloration, which subsequently disappears, ruthenium being deposited at the same time in the metallic state.

Nitrite of potassium colors the solution yellow, with the formation of a double salt, which is readily soluble in water and alcohol. The alkaline solution of this double salt, when mixed with a little colorless sulphide of ammonium, turns crimson (characteristic); on the addition of more sulphide of ammonium, sulphide of ruthenium is precipitated.

Gold.

Hydrosulphuric acid produces in cold solutions a black precipitate of sulphide of gold (Au_2S_3), in boiling solutions a brownish precipitate of the subsulphide (Au_2S). The precipitates are insoluble in hydrochloric and in nitric acid, but dissolve in aqua regia. They are insoluble in colorless sulphide of ammonium, but soluble in yellow sulphide of ammonium, and more readily still in yellow sulphide of sodium.

Sulphide of ammonium precipitates the sulphide of gold.

Ammonia produces in concentrated solutions a reddish-yellow precipitate of aurate of ammonium (fulminating gold). The more acid the solution, and the greater the excess of ammonia, the more gold remains in solution.

Ferrous sulphate precipitates metallic gold in the form of a very finely divided brown powder. The fluid in which the powder is suspended appears dark blue by transmitted light. The dried precipitate shows metallic lustre when pressed with the blade of a knife.

Nitrite of potassium precipitates metallic gold from highly dilute solutions.

Stannous chloride, mixed with stannic chloride, produces in very dilute solutions a precipitate which varies in color, and is sometimes purple (purple of Cassius). The precipitate is insoluble in hydrochloric acid.

Platinum.

Hydrosulphuric acid throws down, after the lapse of some time, a dark brown precipitate of sulphide of platinum (PtS_2). If the solution is heated after the addition of the hydrosulphuric acid, the precipitate forms immediately. It dissolves in a great excess of alkaline sulphides, especially if they contain excess of sulphur. Sulphide of platinum is insoluble in hydrochloric and in nitric acid, but it dissolves in aqua regia.

Sulphide of ammonium produces the same precipitate.

Chloride of ammonium produces, in solutions of chloride of platinum, a yellow crystalline precipitate of $(NH_4)_2PtCl_6$, which is not more soluble in acids than in water. This precipitate is not formed in dilute solutions until the fluid is evaporated to dryness, and the residue is treated with a little water or dilute spirit. Upon ignition the precipitate leaves spongy platinum behind.

Chloride of potassium produces in solution of the chloride a yellow crystalline precipitate of K_2PtCl_6, which has the same properties as the analogous ammonium compound, with the exception that on ignition it leaves behind platinum and chloride of potassium. The decomposition on ignition is not complete, unless the ignition is effected in a current of hydrogen, or with the addition of oxalic acid.

Stannous chloride imparts to solutions of platinum containing much free hydrochloric acid an intensely dark brownish-red color, owing to the reduction of the chloride of platinum to protochloride. No precipitate is formed.

Ferrous sulphate does not precipitate chloride of platinum except upon long-continued boiling, in which case the platinum ultimately suffers reduction.

Iridium.

Iridium is found in combination with platinum and other metals in platinum ores, but more especially as a native alloy of osmium and iridium. Iridium resembles platinum, but is brittle; it fuses with extreme difficulty. In the compact state, or reduced at a red heat by hydrogen, it dissolves in no acid, not even in aqua regia (here it differs from gold and platinum); reduced in the wet way, or largely alloyed with platinum, it dissolves in aqua regia to tetrachloride. Acid sulphate of potassium in a state of fusion will oxidize but not dissolve it (here it differs from rhodium). It oxidizes by fusion with hydrate of sodium with access of air, or by fusion with nitrate of sodium. The compound of sesquioxide of iridium with soda, which is formed in this process, dissolves partially in water; by heating with aqua regia it gives a dark reddish solution of tetrachloride of iridium and chloride of sodium. If iridium in powder is mixed with chloride of sodium, heated to incipient redness and treated with chlorine gas, tetrachloride of iridium and chloride of sodium is formed, which dissolves in water to a deep reddish-brown fluid. Potash added in excess turns the color greenish, while some dark brown tetrachloride of iridium and chloride of potassium (K_2IrCl_6) falls. If the solution is heated, and exposed some time to the air, it acquires at first a deep reddish tint, which changes afterwards to azure (here iridium differs from platinum); if the solution is now evaporated to dryness, and the residue is treated with water, a colorless fluid is obtained, while a blue deposit of binoxide is left undissolved. The solution of the tetrachloride gives the following reactions: —

Hydrosulphuric acid in the first place decolorizes the solution, with the formation of bichloride, sulphur being separated; finally brown sulphide of iridium precipitates.

Sulphide of ammonium produces the same precipitate, which redissolves readily in excess of the precipitant.

Chloride of potassium precipitates tetrachloride of iridium and chloride of potassium (K_2IrCl_6) as a dark brown powder, insoluble in a concentrated solution of chloride of potassium.

Chloride of ammonium precipitates from concentrated solutions tetrachloride of iridium and chloride of ammonium in the form of a dark red powder, consisting of microscopic octahedrons, insoluble in concentrated solution of chloride of ammonium. This double salt (and also the corresponding potassium compound), especially when in hot solution, is turned olive-green by nitrite of potassium, owing to the formation of a double salt of the terchloride of iridium, which on cooling crystallizes out; thus

$$K_2IrCl_6 + KNO_2 = K_3IrCl_6 + NO_2.$$

On heating or evaporating the green solution with an excess of nitrite of potassium it turns yellow, and when boiled deposits a white precipitate which is hardly soluble in water and hydrochloric acid. (This reaction may be taken advantage of to separate the metal from platinum.) If the tetrachloride of iridium and chloride of ammonium is dissolved in water by boiling, and oxalic acid is added, a reduction of the tetrachloride to terchloride takes place, and on this account the solution remains clear on cooling (here iridium differs from platinum).

If *stannous chloride* is added and the solution is boiled, and then excess of potash is added and the mixture is boiled again, a leather-colored precipitate is formed.

Ferrous sulphate decolorizes the solution, but does not produce a precipitate.

Zinc precipitates black metallic iridium.

Molybdenum.

Molybdenum occurs principally as sulphide of molybdenum, and as molybdate of lead. It is tin-white and somewhat malleable; when heated in the air it oxidizes; it is soluble in nitric acid and very difficult to fuse. The protoxide and sesquioxide are black, the binoxide is brown; when heated in the air or treated with nitric acid they are all converted into molybdic anhydride (MoO_3). The latter is a white porous mass, which in water separates into fine scales; it fuses at a red heat; in close vessels it volatilizes only at a very high temperature; in the air it volatilizes easily at a red heat, subliming to transparent laminæ and needles. The non-ignited anhydride dissolves in acids, and also in alkalies. From concentrated solutions in alkalies it is thrown down by nitric acid or hydrochloric acid, but redissolves upon further addition of the precipitant. Molybdic acid gives the following reactions:—

Hydrosulphuric acid, when passed for a long time into the warm acidified solution, ultimately precipitates the whole of the metal as brownish-black tersulphide. The precipitate is soluble in sulphides of the alkali metals, and is reprecipitated on addition of an acid and heating. When ignited in the air, or heated with nitric acid, it is converted into molybdic anhydride.

Zinc or *tin* added to the acidified solution turns it first blue, then green, and ultimately black, with separation of protoxide of molybdenum.

Ferrocyanide of potassium produces a reddish-brown precipitate.

Sulphocyanide of potassium, if added to the solution containing hydrochloric acid, produces no color until zinc is added, when the fluid becomes crimson. Phosphoric acid does not destroy the color (here molybdenum differs from triad iron). On shaking the red fluid with ether, the latter becomes colored.

For the deportment with *phosphoric acid and ammonia,* see p. 89.

Tellurium.

Tellurium is found in the native state, or alloyed with other metals, or as tellurous anhydride (TeO_2). It is a white, brittle, readily fusible metal, and may be sublimed in a glass tube. Heated in the air it burns with a greenish-blue flame, emitting dense white fumes of tellurous anhydride. Tellurium is insoluble in hydrochloric acid, but dissolves readily in nitric acid to tellurous acid (H_2TeO_3). Tellurium in powder dissolves in cold concentrated sulphuric acid to a purple-colored fluid, from which it separates again upon addition of water. Tellurous anhydride is white; at a gentle heat it fuses to a yellow fluid;

it is volatilized by strong ignition in the air, but does not form a crystalline sublimate. It dissolves readily in hydrochloric acid, sparingly in nitric acid, freely in potash, slowly in ammonia, barely in water. Tellurous acid is white; it is perceptibly soluble in cold water, and dissolves in hydrochloric and in nitric acid. By fusing tellurium or tellurites with nitrates and carbonates of the alkali metals, telluric anhydride (TeO_3) is formed, and the fused mass is soluble in water; the solution remains clear upon acidifying with hydrochloric acid in the cold, but upon boiling chlorine is disengaged, tellurous acid is formed, and the solution is now precipitated by water if the excess of acid is not too great. If tellurium, its sulphide, or any of its oxides is fused with cyanide of potassium in a current of hydrogen, a double cyanide of tellurium and potassium is formed; the fused mass dissolves in water, but a current of air throws down from the solution the whole of the tellurium (means of separating tellurium from selenium.) By fusing tellurous or telluric anhydride with carbonate of potassium on charcoal, telluride of potassium is formed, which with acids evolves stinking telluretted hydrogen gas. The solutions of tellurous acid give the following reactions.

Water throws down the acid. From the nitric acid solution nearly the whole of the tellurous acid separates after some time as a crystalline precipitate, even without addition of water.

Hydrosulphuric acid produces in acid solutions a brown precipitate of TeS_2, which resembles stannous sulphide in color. It is freely soluble in sulphide of ammonium.

Potash and *carbonate of sodium* throw down from the hydrochloric acid solution a white precipitate of tellurous acid soluble in excess of the precipitants.

Sulphite of sodium, stannous chloride, and *zinc* throw down black metallic tellurium.

Selenium.

Selenium occurs in nature in the form of metallic selenides. It is found occasionally in the flue dust of roasting furnaces, and also in Nordhausen oil of vitriol. It resembles sulphur in some respects and tellurium in others. Fused selenium is greyish-black; it volatilizes at a high temperature, and may be sublimed. Heated in the air, it burns to selenious anhydride (SeO_2), exhaling a characteristic smell of decaying horse-radish. Concentrated sulphuric acid dissolves selenium without oxidizing it; upon diluting the solution the selenium falls down in red flakes. Nitric acid and aqua regia dissolve selenium to selenious acid (H_2SeO_3). Selenious anhydride is converted at about 200° into a deep yellow gas, which recondenses in the form of white four-sided needles. Both the anhydride and acid are readily soluble in water to a strongly acid fluid. Of the neutral selenites, only those of the alkali metals are soluble in water. All selenites dissolve readily in nitric acid, with the exception of the selenites of lead and silver, which dissolve with difficulty. When selenium or any of its compounds is heated with a carbonate and nitrate of an alkali metal, a seleniate is formed, and the fused mass dissolves in water; upon acidifying with hydrochloric acid, the solution remains clear, and upon then boiling, chlorine is evolved and the selenic acid is reduced to selenious acid. By fusing selenium or any of its compounds with cyanide of potassium in a current of hydrogen, a double cyanide of selenium and potassium is formed, from which the selenium is not eliminated by the action of the air (as is the case with tellurium); it separates, however, upon long-continued boiling after addition of hydrochloric acid. When exposed to the reducing flame on charcoal, selenium compounds evolve the odor of decaying horse-radish (most characteristic). Solutions of selenites show the following reactions:—

Hydrosulphuric acid produces in the presence of hydrochloric acid a yellow precipitate which upon heating turns reddish yellow, and is soluble in sulphide of ammonium. It is doubtful whether this precipitate is a sulphide of selenium.

Stannous chloride and *sulphurous acid* produce in acidified solutions a red precipitate of selenium, which turns grey at high temperatures.

Chloride of barium produces in neutral solutions a white precipitate of selenite of barium, soluble in hydrochloric acid.

Uranium.

This metal is found in a few minerals, as pitchblende, uranochre, &c. It forms two oxides, the uranous (UO), and the uranic (U_2O_3). The uranous oxide is brown, and dissolves in nitric acid to uranic nitrate. The uranic hydrate is yellow; at about 300° it loses its water and turns red; it is converted by ignition into the dark-green urano-uranic oxide. Borax dissolves uranium compounds to yellowish-green or green beads. Solutions of uranium show the following reactions :—

Hydrosulphuric acid fails to produce a precipitate.

Sulphide of ammonium produces in neutral solutions a slowly subsiding precipitate, which is readily soluble in acids, even acetic acid. The precipitation is promoted by chloride of ammonium. The precipitate, when formed in the cold, is chocolate-brown, and contains oxysulphide of uranium, sulphide of ammonium, and water. It is insoluble in yellow sulphide of ammonium; but, when free from other sulphides, it dissolves to a notable extent in colorless sulphide of ammonium, forming a black fluid. On being washed, the precipitate is gradually converted into yellow uranic hydrate. On warming or boiling the mixture of uranium solution and sulphide of ammonium the oxysulphide at first thrown down splits into sulphur and black uranous oxide, which last is insoluble in the excess of sulphide of ammonium. The oxysulphide of uranium (but not the precipitate which has been converted into uranous oxide and sulphur) dissolves readily in carbonate of ammonium. (This reaction may be used as a means of separating uranium from zinc, manganese, iron, &c.) If the oxysulphide remains long in contact with the fluid from which it was precipitated, it gradually turns blood red.

Ammonia and *potash* produce a yellow precipitate of uranic oxide combined with alkali, which is insoluble in excess of the precipitant.

Carbonate of ammonium produces a yellow precipitate of double uranic carbonate with ammonium carbonate, which readily dissolves in excess of the precipitant. Potash throws down the whole of the uranium from a solution formed in this way.

Carbonate of barium precipitates uranium completely in the cold. (Means of separating the metal from nickel, cobalt, manganese, and zinc.)

Ferrocyanide of potassium produces a reddish-brown precipitate. (Very sensitive reaction.)

Indium.

This metal has been found in the blende of Freiberg, and the zinc prepared from the same. The oxide is straw-colored, it does not color the borax bead; when ignited in hydrogen or with charcoal, it is readily reduced, and if a flux be used metallic globules will be obtained. The ignited oxide dissolves slowly in acids in the cold, but readily in the heat. The salts are colorless, the sulphate, nitrate and chloride dissolve readily in water. The chloride is volatile and hygroscopic. Indium tinges flame bluish violet. The spectrum has two characteristic blue lines. With the chloride the lines appear brightest, but they are very transient. For obtaining more persistent lines the sulphide is the most suitable compound.

Hydrosulphuric acid produces no precipitate in the presence of a strong acid. From a solution acidified with acetic acid this reagent throws down sulphide of indium in the form of a slimy precipitate of a fine yellow color. Sulphide of indium is insoluble in cold, but soluble in hot, sulphide of ammonium; on cooling it separates from the solution with a white color.

Sulphide of ammonium added to a solution mixed with tartaric acid and ammonia produces a white precipitate which probably consists of sulphide of indium and hydrogen. It turns yellow on treatment with acetic acid.

Alkalies throw down the hydrate in the form of a white bulky precipitate, like hydrate of aluminium, which is completely insoluble in potash and ammonia. Tartaric acid prevents the precipitation.

Alkaline carbonates precipitate a white gelatinous carbonate. When recently thrown down the precipitate dissolves in carbonate of ammonium, but not in carbonate of potassium or carbonate of sodium. If the solution in carbonate of ammonium is boiled, the carbonate of indium separates again.

Phosphate of sodium throws down a white bulky precipitate.

Alkaline oxalates produce a crystalline precipitate.

Acetate of sodium added to the nearly neutral solution of the sulphate throws down on boiling a basic sulphate.

Carbonate of barium precipitates the whole of the indium, on digestion in the cold. (Means of separating indium from zinc, manganese, cobalt, nickel, and dyad iron.)

Ferrocyanide of potassium produces a white precipitate.

Ferricyanide, sulphocyanide, and chromate of potassium produce no precipitate.

Zinc precipitates the metal in the form of white shining laminæ.

Beryllium.

Beryllium is found in the form of a silicate in phenacite, and with other silicates in beryl, euclase, &c. The oxide is a white tasteless powder insoluble in water. After ignition it dissolves slowly but completely in acids ; it is readily soluble after fusion with acid sulphate of potassium. The hydrate dissolves readily in acids. The compounds of beryllium very much resemble the aluminium compounds. The soluble salts have a sweet astringent taste. The native silicates are completely decomposed by fusion with carbonate of potassium and sodium. Oxide of beryllium when fused with two parts of acid fluoride of potassium, dissolves in water acidified with hydrofluoric acid. (This reaction serves as a means of separating beryllium from aluminium, for when alumina is similarly treated it remains insoluble as fluoride of aluminium and potassium.) The solutions of beryllium salts show the following reactions :—

Ammonia and *sulphide of ammonium* throw down white flocculent hydrate, which is insoluble in ammonia. Upon continued ebullition with chloride of ammonium, the freshly precipitated hydrate dissolves as chloride of beryllium with expulsion of ammonia. (Here the metal differs from aluminium.)

Potash throws down the hydrate, which dissolves in excess of the precipitant. From this solution it is reprecipitated by chloride of ammonium. The concentrated solution in potash remains clear on boiling ; from a dilute solution in potash the whole of the beryllium separates upon continued ebullition. (Here the metal differs from aluminium.)

Carbonate of sodium and *carbonate of ammonium* precipitate white carbonate of beryllium, which redissolves in a large excess of the former and in a much less considerable excess of the latter. (Most characteristic difference between this metal and aluminium.) Upon boiling these solutions, basic carbonate of beryllium separates, readily and completely from the solution in carbonate of ammonium, but only upon dilution and imperfectly from the solution in carbonate of sodium.

Carbonate of barium precipitates beryllium incompletely in the cold, completely upon boiling.

Oxalic acid and *oxalate of ammonium* do not precipitate beryllium. (Here it differs from thorium, zirconium, yttrium, erbium, terbium, cerium, lanthanium, and didymium.)

Thorium.

This metal is found in thorite and monacite. The oxide is white. The ignited oxide is soluble upon heating with a mixture of equal parts of strong sulphuric acid and water; it is not soluble in other acids, even after it has been fused with alkalies. The moist hydrate dissolves readily in acids, the dried hydrate only with difficulty. When chloride or nitrate of thorium are evaporated with hydrochloric or nitric acid respectively, the salts are left in a varnish-like form, and dissolve at once in water completely. Hydrochloric and nitric acids precipitate from such solutions chloride or nitrate of thorium; even sulphuric acid may produce a precipitate in the solutions. Chloride of thorium is not volatile. Thorite (silicate of thorium) is decomposed by strong hydrochloric acid. The solutions of thorium salts give the following reactions:—

Potash, ammonia and *sulphide of ammonium* precipitate the hydrate, which is insoluble in excess of either reagent, even of potash. (Here the metal differs from beryllium.)

Carbonate of sodium and *carbonate of ammonium* precipitate basic carbonate of thorium, which readily dissolves in excess of the precipitant in concentrated solutions, but dissolves with difficulty in dilute solutions. (Here the metal differs from aluminium.) From the solution in carbonate of ammonium, basic salt separates again, even at 50°.

Carbonate of barium precipitates thorium completely.

Hydrofluoric acid precipitates the fluoride which at first appears gelatinous, but after a little while pulverulent. The precipitate is insoluble in water and hydrofluoric acid. (Here the metal differs from aluminium, beryllium, zirconium, and titanium.)

Oxalic acid produces a white precipitate (here the metal differs from beryllium and aluminium). The precipitate does not dissolve in oxalic acid nor in dilute mineral acids, but it does dissolve in a solution of acetate of ammonium containing free acetic acid. (Here the metal differs from yttrium and cerium.)

Sulphate of potassium in concentrated solution precipitates thorium slowly but completely (here the metal differs from aluminium and beryllium.) The precipitate consists of sulphate of potassium and thorium; it is insoluble in concentrated solution of sulphate of potassium; it dissolves with difficulty in cold and also in hot water, but readily on addition of some hydrochloric acid. On heating the neutral solution in cold water, sulphate of thorium separates in the form of a heavy white curdy precipitate. (Here the metal differs from aluminium and beryllium.) This precipitate redissolves in cold water (in which it differs from titanic acid).

Hyposulphite of sodium precipitates from neutral or slightly acid solutions on boiling hyposulphite of thorium mixed with sulphur; the precipitation, however, is not quite complete. (Here the metal differs from yttrium, erbium, terbium, and didymium.)

Zirconium.

This metal is found in zircon and other rare minerals. The oxide is a white powder, insoluble in hydrochloric acid; after continued heating with a mixture of two parts of strong sulphuric acid and one of water it dissolves in water. The hydrate resembles hydrate of aluminium; it dissolves readily in hydrochloric acid when precipitated cold, and still moist, but with difficulty when precipitated hot, or after drying. The native silicates are decomposed by fusion with four parts of carbonate of sodium; the fused mass gives to water silicate of sodium, a sandy zirconate of sodium being left behind, which, after washing, dissolves in hydrochloric acid. Zircon may easily be decomposed by fusion with acid fluoride of potassium at a red heat, fluosilicate of potassium and double fluoride of zirconium and potassium being produced. The solutions of zirconium salts give the following reactions:—

Potash, ammonia and *sulphide of ammonium* precipitate a flocculent hydrate, which is insoluble in excess of either precipitant, even of potash (here zirconium differs from aluminium and beryllium) and is not dissolved by boiling solution of chloride of ammonium (here the metal differs from beryllium).

Carbonate of sodium and *carbonate of ammonium* throw down carbonate of zirconium as a flocculent precipitate, which redissolves in a large excess of carbonate of sodium, and more readily in excess of carbonate of ammonium (here the metal differs from aluminium). On boiling the carbonate of ammonium solution, the zirconium is reprecipitated.

Oxalic acid produces a bulky precipitate of oxalate of zirconium (here the metal differs from aluminium and beryllium). The precipitate is insoluble in oxalic acid, but soluble in hydrochloric acid (here the metal differs from thorium).

Sulphate of potassium in concentrated solution speedily produces a white precipitate of sulphate of zirconium and potassium, which is insoluble in excess of the precipitant (here the metal differs from aluminium and beryllium). The precipitate, if thrown down in the cold, dissolves readily in a large quantity of hydrochloric acid; if thrown down in the heat, it is almost absolutely insoluble in water and in hydrochloric acid (here the metal differs from thorium and cerium.)

Carbonate of barium does not precipitate zirconium completely, even upon boiling.

Hydrofluoric acid produces no precipitate (here the metal differs from thorium and yttrium).

Hyposulphite of sodium produces a precipitate (here the metal differs from yttrium, terbium, erbium and didymium).

Turmeric paper dipped into a solution slightly acidified with hydrochloric or sulphuric acid, acquires a reddish-brown color after drying (here the metal differs from thorium).

Yttrium.

This metal is found in gadolinite, orthite, yttro-tantalite. The ignited oxide dissolves readily in hydrochloric acid (here the metal differs from zirconium and thorium). The ignited oxide is yellowish-white when pure; when it contains erbium and terbium oxides it is brownish-yellow; when it contains didymium it is light chocolate color. The hydrate is pasty and white, without the slightest tinge of red, it attracts carbonic acid, and dissolves, when fresh, in boiling chloride of ammonium (here the metal differs from aluminium and zirconium). The crystallized salts containing water are bright red, the anhydrous salts are white and have a sweet astringent taste. Anhydrous chloride of yttrium is not volatile (here the metal differs from aluminium, beryllium and zirconium). Yttrium compounds give a clear colorless bead with borax in both the outer and inner flame (here the metal differs from cerium and didymium.) The solutions of the salts exhibit the following reactions.

Ammonia and *sulphide of ammonium* precipitate the hydrate. Presence of a small quantity of chloride of ammonium does not interfere with the precipitation by sulphide of ammonium, but a large excess of chloride of ammonium prevents the precipitation.

Potash produces the same precipitate, which is insoluble in excess of the precipitant (here the metal differs from aluminium and beryllium).

Carbonate of potassium produces a precipitate which dissolves with difficulty in excess of the reagent.

Carbonate of ammonium produces a precipitate which is soluble in excess of the precipitant, but by no means so readily as the corresponding beryllium precipitate. The solution of the hydrate in carbonate of ammonium deposits the whole of the yttrium on boiling; if chloride of ammonium is present at the same time, this is decomposed upon continued heating, with separation of ammonia, and the precipitated oxide of yttrium redissolves as chloride. Saturated solutions of carbonate of yttrium in carbonate of ammonium have a tendency to deposit carbonate of yttrium and ammonium, which should be borne in mind.

Oxalic acid produces a white precipitate (here the metal differs from aluminium and beryllium). The precipitate does not dissolve in oxalic acid, it dissolves with difficulty in hydrochloric acid, and it is partially dissolved by boiling with oxalate of ammonium.

Sulphate of potassium precipitates sulphate of yttrium and potassium. The precipitate, even if thrown down hot, dissolves though slowly, in a large quantity of water (here the metal differs from zirconium); on heating this solution the sulphate of yttrium separates. The sulphate of yttrium and potassium dissolves a little more readily in sulphate of potassium than in water (here the metal differs from thorium, cerium, and didymium); it dissolves still more readily in solutions of ammonium salts.

Carbonate of barium produces no precipitate even upon boiling (here the metal differs from beryllium, thorium, cerium, and didymium).

Hydrofluoric acid produces a precipitate (here the metal differs from aluminium, beryllium, zirconium, titanium). The precipitate is gelatinous, insoluble in water and hydrofluoric acid; before ignition it will dissolve in mineral acids, after ignition it is decomposed only by strong sulphuric acid.

Hyposulphite of sodium does not precipitate yttrium. (Here the metal differs from aluminium, thorium, zirconium, titanium.)

Tartaric acid does not prevent the precipitation by alkalies. (Here the metal differs from aluminium, beryllium, thorium, zirconium.) It precipitates tartrate of yttrium. The precipitation frequently does not take place at once, but when it does take place it is complete.

A cold saturated solution of the sulphate becomes turbid when *heated* to between 30° and 40°; on boiling almost the whole of the salt separates.

Turmeric paper is not altered by the acidified solution of the salts. (Here the metal differs from zirconium.)

Terbium and Erbium.*

These metals usually accompany yttrium. On gradually adding ammonia to a solution containing the three metals, the erbium is first precipitated, then the terbium, and then the yttrium. Delafontaine separates the erbium by fractional precipitation with binoxalate of potassium frequently repeated, and subsequent ignition, thus obtaining a mixture of oxide of erbium, with small quantities of oxide of terbium, and still smaller quantities of oxide of yttrium. This mixture he dissolves in nitric acid, saturates hot with sulphate of potassium, and obtains a precipitate of sulphate of erbium and potassium, which, although soluble in hot water, is not soluble in a

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* Popp considers these metals to be mixtures of yttrium with cerium and didymium. Delafontaine, however, defends Mosander's view, that they are distinct metals. (Ann. d. Chem. u. Pharm., 131, 182; 134, 113; 135, 188.)

saturated solution of sulphate of potassium. After this precipitate has been removed the terbium and yttrium may be separated by taking advantage of the fact that oxalate of terbium and potassium is more difficultly soluble in dilute sulphuric acid than the oxalate of yttrium and potassium.

Erbium.

The oxide of erbium, obtained by igniting the hydrate or nitrate, is dark-yellow, often tending to orange-yellow, and heavy; that obtained by igniting the oxalate is pale-yellow, and finely pulverulent. By long ignition in closed vessels it becomes white, losing a small quantity of oxygen. In strong acids it is slowly soluble with evolution of a little oxygen. The hydrate precipitated hot by excess of alkali is white, gelatinous, and attracts carbonic acid from the air; it decomposes ammonium salts in the heat, and readily dissolves in dilute acids; the salts are red. The basic nitrate is dark-yellow (difference from didymium). Erbium salts give, with borax and microcosmic salt, clear, colorless beads (difference from cerium and didymium). Hyposulphite of sodium does not precipitate erbium solutions. A cold saturated solution of binoxalate of potassium gives, with erbium solutions, after some time, a crystalline precipitate. With regard to the spectroscopic reactions, see Delafontaine ("Ann. d. Chem. u. Pharm.," 135, 191).

Terbium.

Oxide of terbium is yellow, soluble in acids after strong ignition; the hydrate is a white gelatinous precipitate, remaining white on drying. Oxide of terbium expels ammonia from its salts even in the cold, more easily when heat is applied. The salts of terbium are colored darker than those of erbium. The solution of nitrate of terbium is pink, when concentrated darker, but without the slightest tinge of violet (difference from didymium). Terbium behaves like erbium with borax, microcosmic salt, hyposulphite of sodium, and binoxalate of potassium. As regards the spectroscopic reactions see Delafontaine ("Ann. d. Chem. u. Pharm." 135, 195).

Cerium

Occurs in cerite, orthite, &c. There are three oxides—the protoxide (cerous oxide), the sesquioxide (ceric oxide), and the peroxide. The cerous hydrate is white, and turns yellow in the air from absorption of oxygen, by ignition in the air it turns orange color or red and is converted into ceric oxide. The cerous hydrate is readily soluble in acids, the ignited ceric oxide, when it contains lanthanium and didymium oxide, dissolves readily in hydrochloric acid with evolution of chlorine, when pure it scarcely at all dissolves in boiling hydrochloric acid, but it does dissolve on addition of alcohol (difference from thorium and zirconium); the solutions contain cerous chloride. The ceric oxide dissolves in concentrated sulphuric acid, although with difficulty; it is hardly attacked by nitric acid. The ceric oxide obtained from the oxalate when evaporated with nitric acid yields a basic salt, which gives an emulsion with water, and is not completely soluble in very considerable quantities of water (difference from alumina). The cerous salts are colorless, occasionally with a tinge of amethyst; the soluble salts redden litmus. Cerous chloride is non-volatile (difference from aluminium, beryllium, and zirconium). The sulphate deposits a precipitate on solution in boiling water. Cerite is a hydrous silicate of cerous oxide, lanthanium oxide, and didymium oxide, it does not dissolve in aqua regia, but is decomposed by fusion with carbonate of sodium, and is also decomposed by concentrated sulphuric acid.

The cerous salts show the following reactions:—

Ammonia precipitates basic salt insoluble in excess of the precipitant. Tartaric acid prevents the precipitation (here the metal differs from yttrium).

Potash precipitates white hydrate, which becomes yellow in the air. It is insoluble in excess of the precipitant (here the metal differs from aluminium and beryllium). Tartaric acid does not interfere with the precipitation.

Carbonate of potassium produces a white precipitate, slightly soluble in excess of the precipitant.

Carbonate of ammonium produces a white precipitate, rather soluble in excess of the precipitant.

Oxalic acid precipitates the cerium completely, even from moderately acid solutions (here the metal differs from aluminium and beryllium). The precipitate does not dissolve in oxalic acid, but it is soluble in a large quantity of hydrochloric acid.

Carbonate of barium throws down the cerium completely on standing.

Hyposulphite of sodium does not precipitate cerium, even on boiling with very concentrated solutions. The precipitated sulphur only carries down traces of the salt with it.

A saturated solution of *sulphate of potassium* precipitates, even from somewhat acid solutions, white cerous potassic sulphate (here the metal differs from aluminium and beryllium), which is difficultly soluble in cold water, readily soluble in hot water, and insoluble in a saturated solution of sulphate of potassium (here the metal differs from yttrium). The precipitate may be dissolved by boiling with much water containing hydrochloric acid.

If we conduct *chlorine* through a cerous solution, not too acid and mixed with acetate of sodium, or if we add chloride of soda to such a solution, all the cerium is precipitated as light yellow peroxide (free from didymium and lanthanium).

If a cerous salt be dissolved in nitric acid, with addition of an equal volume of water, and if a small quantity of *binoxide of lead* be added, and the liquid be boiled for some minutes, the solution turns yellow, even if only small quantities of cerium be present. On evaporating this solution to dryness, heating the residue till a portion of the acid escapes, and treating it with water acidified with nitric acid, no cerium will be dissolved, but any didymium and lanthanium present will be dissolved.

Solutions of ceric salts are precipitated in the cold by carbonate of barium.

Hyposulphite of sodium precipitates a solution of ceric nitrate.

Lanthanium.

This metal is generally associated with cerium. The oxide is white, and remains unaltered by ignition in the air (here it differs from cerous oxide). The oxide when brought into contact with water is converted into a milk-white hydrate; with cold water the conversion is slow, with hot water rapid. The oxide and hydrate color litmus blue; they dissolve in boiling solution of chloride of ammonium, and also in dilute acids (here lanthanium resembles magnesium). The salts are colorless; the saturated solution of the sulphate in cold water deposits a portion of the salt even at 30° (here the metal differs from cerium). The solutions of lanthanium show the following reactions:—

Ammonia precipitates basic salts, which pass milky through the filter on washing.

Potash precipitates the hydrate, which is insoluble in excess of the precipitant, and does not turn brown in the air.

Carbonate of ammonium produces a precipitate which is insoluble in excess of the precipitant (here the metal differs from cerium).

Sulphate of potassium, oxalic acid, and *carbonate of barium* give the same reactions as with cerous salts.

If a cold dilute solution of acetate of lanthanium is supersaturated with ammonia, the slimy precipitate repeatedly washed with cold water, and a little powdered *iodine* added, a blue coloration makes its appearance, which gradually pervades the whole mixture.

Didymium.

This metal is also associated with cerium. The oxide when brought into contact with water is slowly converted into hydrate, it rapidly attracts carbonic acid, its reaction is not alkaline, it dissolves readily in acids. The concentrated solutions have a reddish or faint violet color. The nitrate, on heating, is first converted into a basic salt (here the metal differs from lanthanium), which is grey. The chloride is non-volatile. The saturated solution of the sulphate deposits salt, not at 30°, but upon boiling. Didymium oxide gives with borax in both flames a nearly colorless bead. Microcosmic salt dissolves the oxide in the reducing flame to an amethyst-red bead, inclining to violet. With carbonate of sodium in the outer flame we obtain a greyish-white bead (here the metal differs from manganese). For the spectroscopic reactions see Erdmann "Journ. f. Prakt. Chem.," 85, 395; and Delafontaine "Ann. d. Chem. u. Pharm.," 135, 195. The solutions of didymium show the following reactions.

Ammonia precipitates a basic salt, which is insoluble in ammonia, but slightly soluble in chloride of ammonium.

Potash precipitates the hydrate, which is insoluble in excess of the precipitant, and does not alter in the air.

Carbonate of sodium and *carbonate of ammonium* produce a copious precipitate, which is insoluble in excess of the precipitant, even in excess of carbonate of ammonium (here the metal differs from cerium), but dissolves slightly in concentrated solution of chloride of ammonium.

Carbonate of barium precipitates didymium slowly (more slowly than cerium and lanthanium), and never completely.

Oxalic acid precipitates didymium almost completely; the precipitate is difficultly soluble in cold hydrochloric acid, but it dissolves in this acid upon application of heat.

Sulphate of potassium in concentrated solution precipitates didymium solutions more slowly and less completely than cerous solutions. The precipitate is insoluble in sulphate of potassium, and also in water; but it is soluble, though with difficulty, in hot hydrochloric acid.

Hyposulphite of sodium produces no precipitate.

For the separation of cerium from lanthanium and didymium, one of the following methods may be used:—*a.* Nearly neutralize the solution of the three metals if acid, without allowing any permanent precipitate to form, add a sufficient quantity of acetate of sodium and an excess of chloride of soda, and boil; the cerium will fall as peroxide, while lanthanium and didymium remain in solution. (Popp, "Ann. d. Chem. u. Pharm.," 131, 360.) *b.* Precipitate the metals with potash, wash, suspend the precipitate in potash, and pass chlorine. Lanthanium and didymium dissolve; the peroxide of cerium remains behind. (Damour and St. Claire Deville, "Compt. Rend.," 59, 272.) *c.* Dissolve in a large excess of nitric acid; boil with peroxide of lead; evaporate the orange-colored solution to dryness, and heat the residue till a portion of the acid escapes; treat with water acidulated with nitric acid, and separate the insoluble basic ceric nitrate from the solution which contains all the lanthanium and didymium. (Gibbs, "Zeitschr. f. anal. Chem.," 3, 396.) In using the last method, before proceeding with the residue or solution, the lead must be first separated by sulphuretted hydrogen.

From the solution of lanthanium and didymium obtained by one or other of the above methods, the metals are precipitated with oxalate of ammonium, the oxalates are ignited, and the oxides thus obtained are treated with dilute nitric acid. If the separation of cerium was incomplete, the remainder of the cerium will here remain behind. The solution is evaporated in a dish with a flat bottom to dryness and heated to 400—500°. The salts fuse; nitrous fumes escape. The residue is treated with hot water, which dissolves the nitrate of lanthanium, leaving behind grey basic nitrate of didymium. By a repetition of the evaporation, &c., the two metals may be satisfactorily separated. (Damour and St. Claire Deville.)

Titanium.

There are two oxides of titanium—oxide of titanium and titanic anhydride. The latter is more frequently met with in analysis. Titanic anhydride is found in the free state in rutile and anatase, and in combination in titanite, titaniferous iron, &c.; it is also found in small quantities in many iron ores, in clays, in silicates generally, and in blast-furnace slags. The small copper-colored cubes which are occasionally found in such slags consist of cyanide and nitride of titanium. Titanic anhydride when feebly ignited is white; very intense ignition gives a yellowish or brownish tint to it; it is infusible and insoluble in water; its density varies between 3·9 and 4·25. Ignited titanic anhydride is insoluble in acids, except the hydrofluoric and the strong sulphuric acid. If the solution in hydrofluoric acid is evaporated with addition of sulphuric acid, no fluoride of titanium will volatilize. (Here titanic anhydride differs from silicic anhydride.) When titanic anhydride is fused for a long time with acid sulphate of potassium, a clear mass is obtained, which dissolves in a large proportion of cold water to a clear fluid. (Hydrous) titanic acid dissolves when moist and when dried without heat, in dilute acids, especially in hydrochloric and sulphuric acids. If titanic anhydride is fused with potash, and the fused mass is treated with water, the solution will contain a little titanic acid. When titanic anhydride is fused with the carbonates of potassium and sodium, carbonic acid is expelled, and upon treating the fused mass with water, alkali and alkaline carbonate are dissolved, while acid titanate of alkali metal is left behind and may be dissolved in hydrochloric acid. Titanic anhydride mixed with charcoal and heated in a current of chlorine gives tetrachloride of titanium as a volatile liquid emitting copious fumes in the air. Microcosmic salt readily dissolves titanic anhydride in the outer flame to a clear bead, colorless when cold; on exposure to the reducing flame, with addition of a little ferrous sulphate, the bead acquires a blood-red color. Solutions of titanic acid in hydrochloric or sulphuric acid show the following reactions:—

When the solution (especially the solution in sulphuric acid) is subjected in a highly dilute state to long-continued *boiling*, the titanic acid is separated as a white powder, insoluble in dilute acids. Presence of much free acid retards the separation and diminishes the quantity of the precipitate. The precipitate which separates from a hydrochloric acid solution may be filtered off, but it will pass milky through the filter upon washing, unless an acid or chloride of ammonium is added to the washing water.

Ammonia and *sulphide of ammonium* throw down titanic acid as a white bulky precipitate, insoluble in excess of either of the precipitants. When thrown down cold and washed with cold water, it is soluble in hydrochloric acid and dilute sulphuric acid. Presence of tartaric acid prevents its formation.

Potash produces the same precipitate, insoluble in excess of the precipitant.

Carbonate of barium also throws down titanic acid.

Ferrocyanide of potassium produces a dark brown precipitate.

Hyposulphite of sodium boiled with solution of titanic acid precipitates all the titanium.

Zinc or *tin* produces after some time a pale violet or blue coloration; subsequently a blue precipitate, which gradually becomes white. The coloration is caused by the reduction of the titanic acid to oxide of titanium. If to the blue but still clear solution potash or ammonia is added, blue hydrate of titanium separates, which is gradually converted into white titanic acid with decomposition of water.

The solution of the tetrachloride of titanium in cold water is changed by heat. The solution prepared with cold water is not precipitated by sulphuric acid, nor by hydrochloric, nor by nitric, it is precipitated by phosphoric acid, arsenic acid, or iodic acid; but if the solution be boiled only for a few seconds it becomes slightly opalescent, and so far modified that hydrochloric and nitric acids produce white precipitates in it which are insoluble in excess of the acids, sulphuric acid also precipitates it, but an excess redissolves the precipitate. The solution prepared in the cold contains titanic acid, the boiled solution contains metatitanic acid. The difference also attaches to the acids. (R. Weber, "Pogg. Ann." 120, 287.)

Tantalum.

Tantalum forms two oxides, oxide of tantalum and tantalic anhydride. The latter is found in tantalite, yttrotantalite, columbite, and some other rare minerals. Tantalic anhydride is white; after ignition its density is about 7 or 8; it combines with acids and bases. The ignited tantalic anhydride is insoluble in hydrochloric acid and in strong sulphuric acid; when it is fused with acid sulphate of potassium and the mass is extracted with water, the tantalic anhydride is left behind in combination with sulphuric acid. In this last reaction, tantalic anhydride differs from titanic anhydride, but they cannot be well separated from each other by this means. When the compound of sulphuric acid and tantalic anhydride is ignited in an atmosphere of carbonate of ammonium, pure tantalic anhydride is left behind. Tantalic anhydride when fused with potash for a long time gives a mass soluble in water; the solution contains tantalate of potassium. By fusing tantalic anhydride with soda, a turbid mass is obtained; if this mass is treated with water, the excess of soda will be dissolved, and the whole of the tantalate of sodium will be left behind, as it is insoluble in solution of soda; however, after the removal of the excess of soda, the tantalate of sodium will dissolve in water. By igniting tantalic anhydride with charcoal in a current of pure dry chlorine, yellow chloride of tantalum is formed, which sublimes in crystals; if the tantalic anhydride contains titanic anhydride, tetrachloride of titanium is formed at the same time, which emits copious fumes in the air.

Microcosmic salt dissolves tantalic anhydride to a colorless bead, which does not acquire a red tint on addition of ferrous sulphate (here tantalic differs from titanic anhydride). The solutions of tantalic acid show the following reactions:—

Hydrochloric acid added to solution of alkaline tantalate gives a precipitate, which redissolves in excess of the reagent to an opalescent fluid.

Ammonia and *sulphide of ammonium* throw down from a solution of alkaline tantalate, acidified with hydrochloric acid, tantalic acid or acid tantalate of ammonium. Presence of tartaric acid prevents the precipitation.

Soda throws down from solution of tantalate of sodium, the latter salt; if the reagent is added slowly, the precipitate will be crystallized.

Sulphuric acid throws down from solutions of alkaline tantalates, sulphate of tantalic anhydride.

Carbonic acid throws down from solutions of alkaline tantalates, acid salts, which are not dissolved by boiling with carbonate of sodium.

Ferrocyanide of potassium added to an acidified solution throws down a yellow precipitate.

Zinc added to solution of alkaline tantalate, strongly acidified with hydrochloric acid, produces no coloration, even after the addition of sulphuric acid. But if solid chloride of tantalum is dissolved in strong sulphuric acid, and water and zinc are added to the solution, the fluid will turn a blue color, which will not change to brown on standing.

Vanadium.

Vanadium occurs in the form of vanadiates, occasionally in small quantities in iron and copper ores, and in the slags obtained from the same. There are several oxides. The highest oxide is the anhydride of vanadic acid; it is yellowish red, and fuses at an incipient red heat, solidifying on cooling to a crystalline mass; it is non-volatile. It is very difficultly soluble in water, but reddens moist litmus paper strongly. It combines with bases and acids. Strong acids dissolve it to yellow or red fluids. The solutions are often decolorized by boiling. Most of the neutral vanadiates are yellow, those of the alkaline metals, and some others, are converted into a colorless modification by heating with water. Most of the salts are soluble in water, all are soluble in nitric acid. The solutions of vanadic acid show the following reactions.

Hydrosulphuric acid produces no precipitate in acid solutions, but a blue color accompanied by separation of sulphur.

Sulphide of ammonium added to a solution acidified with sulphuric acid precipitates brown vanadic sulphide, which dissolves in excess of the precipitant to a reddish brown fluid.

Chloride of ammonium added to a solution of an alkaline vanadiate precipitates the whole of the vanadium as white vanadiate of ammonium, insoluble in chloride of ammonium. (Most characteristic reaction.) The precipitate, on ignition, gives vanadic anhydride, or a mixture of the same with a lower oxide.

Peroxide of hydrogen, added to an acidified solution of an alkaline vanadiate gives, on shaking, a red color. On adding ether, and again shaking the mixture, the solution retains its color, and the ether remains colorless. (Most delicate reaction.)

Ferrocyanide of potassium produces, in acid solution, a green flocculent precipitate, insoluble in acids. In solutions of vanadiates it produces a yellow precipitate.

Sulphurous acid, stannous chloride, many metals, organic substances, &c., reduce vanadic acid in acid solution, and color the fluid green or blue.

Nitrate of silver and *chloride of barium* produce white or yellow precipitates in solutions of vanadiates. The precipitates are readily soluble in acids.

Lithium.

Lithium is found widely disseminated in nature, but in minute quantities only. It is often met with in the analysis of mineral waters and ashes of plants, less frequently in the analysis of minerals. The oxide dissolves with difficulty in water; it does not attract moisture from the air. Most of the salts are soluble in water; the chloride is deliquescent. The carbonate is difficultly soluble, particularly in cold water. The salts tinge flame carmine. Chloride of platinum and tartaric acid fail to precipitate even strong solutions of the salts. Phosphate of sodium produces in solutions of lithium salts, upon boiling, a white crystalline precipitate of $2Li_3PO_4+H_2O$, which quickly subsides. This reaction, which is characteristic of lithium, is rendered much more delicate by adding with the phosphate of sodium a little soda, just sufficient to leave the reaction alkaline, evaporating to dryness, treating the residue with water, and adding an equal volume of ammonia. In this manner very minute quantities of lithium will be separated in the above form. If the precipitate is dissolved in hydrochloric acid, and the solution is supersaturated with ammonia, it remains clear in the cold, but upon boiling a heavy crystalline precipitate of the above composition falls.

Cæsium and Rubidium.

The compounds of these metals are pretty widely disseminated, but they are found invariably in very minute quantities only. They have been detected in the mother liquors of mineral waters, and in a few minerals (melaphyr, carnallite, lepidolite). Cæsium has been found in considerable quantities in pollux, and traces of rubidium have been found in the ashes of plants. The compounds of cæsium and rubidium bear in general great resemblance to the potassium compounds; their strong solutions are precipitated by chloride of platinum, and those which are volatile color flame violet. On the other hand, the platinum salts are far less soluble in water than the corresponding salt of potassium; 100 c.c. water at 10° dissolves 900 mgrm. of the chloride of potassium and platinum, 154 mgrm. of the chloride of rubidium and platinum, and 50 mgrm. of the chloride of cæsium and platinum. Again, the alums show great differences as regards their solubility in cold water; thus 100 parts of water at 17° dissolve 13·5 parts of potassium alum, 2·27 parts of rubidium alum, and ·619 parts of cæsium alum. And again, the spectra of cæsium and rubidium are quite different from the spectrum of potassium.

The carbonate of cæsium is soluble in absolute alcohol, while carbonate of rubidium is insoluble. However, the two metals cannot well be separated by this means, as the carbonates seem to form a double salt, which is not absolutely insoluble in alcohol. It is more easy to separate them when they are in the form of acid tartrates; the acid tartrate of rubidium dissolves in 8·5 parts of boiling water, and 84·57 parts of water at 25°, while the corresponding salt of cæsium dissolves in 1·02 parts of boiling water, and 10·32 parts of water at 25°. (The acid tartrate of potassium requires 15 parts of boiling water, and 89 parts of water at 25°.)

DETECTION OF THE RARE METALS IN THE COURSE OF ANALYSIS.

The precipitate produced by Hydrochloric Acid

May contain *silver, mercury, lead,* thallium (as chlorides), niobous acid, tungstic acid, and *silicic acid.* Antimonic, tantalic, and molybdic acids are also precipitated, but redissolve in excess of hydrochloric acid; tantalic acid redissolves to an opalescent fluid.*

The precipitate is thoroughly washed with hot water; and the washings are tested for **thallium** with iodide of potassium.

The residue is treated with ammonia to remove the silver, and then with nitric acid to remove the mercury. Niobous acid, tungstic acid, and silicic acid remain behind.

To separate the silica, the residue is fused with acid sulphate of potassium, treated with water, and then with dilute carbonate of ammonium; and to separate the **niobium** from the **tungsten** the solution obtained is treated with excess of sulphide of ammonium.

The precipitate produced by Hydrosulphuric Acid

May contain

1. As sulphides insoluble in sulphide of ammonium, or sulphide of sodium. *Lead, mercury, bismuth, copper, cadmium,* palladium,† rhodium,† osmium,† ruthenium.†
2. As sulphides soluble in sulphide of ammonium, or sulphide of sodium. *Tin, antimony, arsenic,* gold, platinum,† iridium,† molybdenum, tellurium, selenium.‡

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* Hyposulphites are decomposed by hydrochloric acid, sulphur being deposited and sulphurous acid formed.

† These metals are not readily precipitated; the gas must be passed for a long time, and the solution must be warmed.

‡ The following bodies cause separation of sulphur. The salts of man-

After the precipitate has been separated into the two divisions, they are examined as follows :—

Division I.

Is fused with potash and chlorate of potassium, heated finally to redness, allowed to cool, treated with water and filtered.

The filtrate may contain osmate and ruthenate of potassium —the latter gives a deep yellow color. It is cautiously neutralized with nitric acid, when black sesquioxide of **ruthenium** will separate. On adding excess of nitric acid and distilling, the **osmium** will pass over in the form of osmic acid.

The residue is gently ignited in hydrogen,[*] then cautiously treated with dilute nitric acid. The copper, lead, &c., are dissolved, while the rhodium and palladium remain. The residue is treated with aqua regia, which dissolves the **palladium,** leaving the **rhodium.**

A separate portion of the precipitate of Division I. should be tested for mercury in this case.

Division II.

On fusing the precipitate with carbonate and nitrate of sodium, according to p. 72, and treating it with cold water, the solution will contain **molybdic, telluric, and selenic acids,** besides arsenic acid, while **gold, platinum,** and **iridium** will be left behind in the metallic state with the stannic oxide and antimoniate of sodium.

The precipitate produced by Ammonia and Sulphide of Ammonium in presence of Chloride of Ammonium

May contain

As sulphides. *Nickel, cobalt, iron, manganese, zinc,* uranium, thallium, indium.

As hydrates. *Chromium, aluminium,* beryllium, thorium,

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ganese and cobalt corresponding to their higher oxides, vanadic acid (with blue coloration), and nitrous, sulphurous, hyposulphurous, bromic, iodic acids.

[*] Cadmium may escape in this operation.

zirconium, yttrium, terbium, erbium, cerium, lanthanium, didymium, titanium, tantalum, (niobium.*)

1. The greater part of the precipitate is washed, dried, ignited in a porcelain crucible, fused for a long time in a platinum crucible with acid sulphate of potassium, allowed to cool, well soaked in cold water and filtered.

The residue contains the **tantalum,** niobium, and possibly silica and a little undissolved iron and chromium. It is fused with soda and chlorate of potassium, and treated with dilute soda; chromate and silicate of potassium will dissolve, while ferric oxide, tantalate and niobite of sodium will be left. The excess of soda is removed from the residue, and then the latter is treated repeatedly with very dilute carbonate of sodium, in which the niobite of sodium dissolves much more readily than the tantalate.

The filtrate is treated with hydrosulphuric acid, to reduce triad iron, much diluted, and boiled for some time, carbonic acid being passed all the while. A precipitate may contain **titanium,** and possibly zirconium.

The filtrate is evaporated to a small bulk, with addition of nitric acid, mixed with excess of ammonia, and filtered. The precipitate is washed, dissolved in hydrochloric acid, reprecipitated with ammonia, and filtered off; the filtrate being mixed with the former filtrate. The mixed filtrates contain almost the whole of the nickel, cobalt, manganese, and zinc.

The precipitate is redissolved in hydrochloric acid, mixed in the cold with potash, and filtered.

The filtrate contains the chromium, aluminium, and beryllium. It is diluted and boiled for some time; the aluminium will remain in solution, the beryllium and chromium will be precipitated. The precipitate is fused with carbonate of sodium and chlorate of potassium in a platinum crucible and boiled with water. All the chromium will dissolve as chromate. The **beryllium** may partly remain insoluble and partly dissolve; the part which dissolves may be thrown down by acidifying with hydrochloric acid and then neutralizing with ammonia.

The precipitate may still contain, besides the other bodies, chromium, and possibly (in presence of yttrium and cerium)

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* Traces of niobium may be found here, which have been left unprecipitated by the hydrochloric acid.

aluminium and beryllium. It is dissolved in hydrochloric acid, evaporated if necessary to drive off excess of acid, diluted, mixed with carbonate of barium, and allowed to stand four or five hours in the cold.

The precipitate contains iron, indium, and possibly chromium, aluminium and uranium. It is redissolved in hydrochloric acid, and mixed with excess of acid carbonate of sodium to separate **uranium.** The precipitate is tested in the spectroscope for **indium,** and by fusing with carbonate of sodium and chlorate of potassium for chromium.

In the filtrate from the carbonate of barium precipitate the barium is thrown down by sulphuric acid, the sulphate of barium is filtered off, the filtrate is evaporated to a small bulk, exactly neutralized with potash (the solution being left rather acid than alkaline), mixed with sulphate of potassium in crystals, boiled, allowed to stand for twelve hours, and filtered; the precipitate being washed with sulphate of potassium.

The filtrate contains yttrium, erbium, terbium, and possibly some beryllium. It is mixed with ammonia, which precipitates all four. The precipitate is treated with a strong, hot solution of oxalic acid, which dissolves the **beryllium** leaving **yttrium, erbium,** and **terbium.**

The precipitate that has been washed with sulphate of potassium is boiled repeatedly with water containing a little hydrochloric acid; the **thorium** and **cerium** will dissolve, leaving the **zirconium** behind.

2. A portion of the precipitate is tested in the spectroscope for **thallium** (and indium). To examine further for thallium, dissolve a portion of the precipitate in boiling dilute hydrochloric acid, treat the solution with sulphurous acid till the ferric salts are reduced, neutralize nearly with ammonia, and add iodide of potassium. If a precipitate is obtained, it should be examined in the spectroscope.

The filtrate from the Sulphide of Ammonium Precipitate.

By just acidifying this filtrate with hydrochloric acid we may get a precipitate containing *nickel,* vanadium, and tungsten,* as sulphides. The precipitate is filtered off, washed,

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* A portion of this metal may have been left by the hydrochloric acid.

dried, fused with carbonate of sodium and nitrate of potassium, and treated with water, which will dissolve vanadiate and tungstate of potassium, leaving oxide of nickel behind. From the solution the **vanadium** may be separated by solid chloride of ammonium, and the **tungsten** by evaporating with hydrochloric acid and treating the residue with water.

Detection of Lithium, Cæsium and Rubidium in presence of large quantities of Sodium and Potassium.

The dry chlorides are extracted, with addition of a few drops of hydrochloric acid,* with alcohol of 90 per cent. The solution will contain all the rare metals, and almost the whole of the potassium and sodium will be left behind. The solution is evaporated to dryness, taken up with a little water, mixed with chloride of platinum and filtered.

The precipitate contains the cæsium and rubidium. It is washed repeatedly with boiling water to remove the chloride of potassium and platinum, and examined continually by the spectroscope during the process. The potassium spectrum will grow fainter and fainter while the spectra of **cæsium** and **rubidium** will become visible.

The filtrate contains the lithium. It is evaporated to dryness, heated to low redness in a current of hydrogen to decompose the chloride of sodium and platinum, and the excess of chloride of platinum, moistened with hydrochloric acid,* warmed to drive off the excess of the acid, and extracted with a mixture of absolute alcohol and ether. On evaporating the alcoholic solution, the chloride of **lithium** is obtained perfectly free from sodium and potassium. It may be well to dissolve a portion in water, and add sulphuric acid and alcohol to make sure that strontium and calcium are absent.

Insoluble Substances

May contain the following rare metals :—Beryllium, thorium, zirconium, cerium, titanium, tantalum, niobium, molybdenum, tungsten, rhodium, iridium, osmium-iridium, ruthenium.

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* The use of the hydrochloric acid is to reconvert any carbonate of lithium that may be formed into chloride. Carbonate of lithium is insoluble in alcohol.

The substance, after having been freed from silver, lead, and sulphur in the usual way, is fused with carbonate of sodium and nitrate of potassium, and repeatedly extracted with hot water. If a residue is left, it is fused for some time in a silver crucible with potash and nitrate of potassium, and treated repeatedly with water.

The alkaline solutions are mixed. They may contain beryllium, a portion of the titanium, tantalum, niobium, molybdenum, tungsten, osmium, ruthenium, and a portion of the iridium.

The residue is fused with acid sulphate of potassium, and treated with water. The solution may contain thorium, zirconium, cerium, the remainder of the titanium, and rhodium. If a residue is still left, it may contain platinum ore metals; it should be mixed with chloride of sodium and ignited in a current of chlorine.

REACTIONS OF RARE ACIDS.

LIST OF RARE ACIDS.

Hydrofluosilicic acid.
Iodic acid.
Nitrous acid.
Perchloric acid.
Pyrophosphoric acid.
Metaphosphoric acid.
Phosphorous acid.
Hypophosphorous acid.
Sulphurous acid.
Hyposulphurous acid.

Hydrofluosilicic Acid.

When evaporated in platinum this acid volatilizes completely as fluoride of silicon and hydrofluoric acid. When evaporated in glass it etches the vessel. The metallic silicofluorides are resolved by ignition into metallic fluorides and fluoride of silicon.

When silicofluorides are heated with *strong sulphuric acid* sulphates are formed, and hydrofluoric acid and fluoride of silicon escape. If the experiment is conducted as described, p. 87, 2, the glass will be etched.

Chloride of barium forms a crystalline precipitate, see p. 59, 8.

Chloride of strontium and *acetate of lead* form no precipitate.

Salts of potassium precipitate transparent gelatinous silicofluoride of potassium.

Ammonia in excess precipitates hydrated silica, fluoride of ammonium being formed.

Iodic Acid.

Chloride of barium throws down from alkaline iodates a white precipitate of iodate of barium, which is soluble in nitric acid.

Nitrate of silver throws down a white crystalline precipitate of iodate of silver, readily soluble in ammonia, sparingly soluble in nitric acid.

Hydrosulphuric acid throws down from iodic acid solutions iodine, which dissolves in the hydriodic acid formed; sulphur being separated at the same time. An excess of hydrosulphuric acid decolorizes the solution, the iodine being completely converted into hydriodic acid.

Sulphurous acid precipitates iodine, which is converted into hydriodic acid by an excess of the reagent.

Nitrous Acid.

When a nitrite is mixed with an *acid,* the nitrous acid which might be supposed to be formed, splits up at once into nitric acid, nitric oxide and water, thus—

$$3\,HNO_2 = HNO_3 + 2\,NO + H_2O.$$

Nitrate of silver added to alkaline nitrites produces a white precipitate, which dissolves in a very large quantity of water, especially of hot water.

Ferrous sulphate upon addition of an acid, produces a dark brown coloration, due to the nitric oxide dissolving in the ferrous solution.

Hydrosulphuric acid produces in neutral solutions a copious precipitate of sulphur, nitrate of ammonium being formed at the same time.

The most delicate test for nitrous acid consists in the addition of *iodide of potassium, solution of starch and sulphuric acid.* Water containing the one hundred-thousandth part of nitrite of potassium is colored distinctly blue in a few seconds, and a few minutes suffice to produce the same effect in water containing one-millionth of the salt. However, this test is not characteristic, as other substances besides nitrous acid have this effect.

Perchloric Acid.

The dilute solution of the acid gives by distillation, first water, then weak acid, and finally strong acid. All the per-chlorates are decomposed by ignition, those of the alkali metals leaving chlorides behind, with disengagement of oxygen.

Salts of potassium produce a white crystalline precipitate of perchlorate of potassium, sparingly soluble in water, insoluble in spirit.

Chloride of barium and *nitrate of silver* produce no precipitate.

Strong sulphuric acid fails to decompose perchloric acid in the cold, and decomposes it only with difficulty on heating. (Here perchloric acid differs from chloric acid.)

Hydrochloric acid, nitric acid and sulphurous acid do not decompose perchloric acid; if therefore indigo solution is added at the same time it is not decolorized. (Here perchloric acid differs from all the other oxygen acids of chlorine.)

Pyrophosphoric Acid.

The solution of this acid when boiled is converted into phosphoric acid. The solutions of the salts may be heated without suffering decomposition. If the salts are fused with carbonate of sodium, phosphates are formed.

Chloride of barium fails to precipitate the free acid; from solutions of its salts pyrophosphate of barium is precipitated.

Nitrate of silver throws down from a solution of the acid, especially on addition of an alkali, a white precipitate of pyrophosphate of silver, which is soluble in nitric acid and ammonia.

Sulphate of magnesium precipitates pyrophosphate of magnesium. The precipitate dissolves in excess of the pyrophosphate, as well as in excess of sulphate of magnesium. Ammonia fails to reprecipitate it from these solutions. Upon boiling the solution it separates again.

A concentrated solution of *luteo-cobaltic chloride* added to an alkaline pyrophosphate produces an immediate precipitation of pale reddish-yellow spangles. (Here pyrophosphoric acid differs from phosphoric and metaphosphoric acids.)

White of egg is not precipitated by pyrophosphoric acid, nor by its salts mixed with acetic acid.

Molybdate of ammonium dissolved in nitric acid, fails to produce a precipitate.

Metaphosphoric Acid.

The salts when fused with carbonate of sodium yield phosphate of sodium.

Those solutions which are precipitated by *nitrate of silver* produce a white precipitate with that reagent.

A mixture of *sulphate of magnesium, chloride of ammonium, and ammonia* fails to produce a precipitate. Or if a precipitate is formed it is soluble in chloride of ammonium.

White of egg is immediately precipitated by metaphosphates acidified with acetic acid.

Phosphorous Acid.

The solution of the acid is decomposed by heat into phosphoric acid and phosphuretted hydrogen gas, which does not take fire spontaneously. All the salts are decomposed by ignition into phosphates and hydrogen, or a mixture of hydrogen and phosphuretted hydrogen.

Nitrate of silver is reduced to metallic silver, more especially upon adding ammonia and heating.

Mercurous nitrate is reduced to metallic mercury, the reaction being favoured by addition of ammonia and heating.

Mercuric chloride is reduced to mercurous chloride, especially on heating.

Chloride of barium and chloride of calcium produce, upon addition of ammonia, white precipitates soluble in acetic acid.

A mixture of *sulphate of magnesium, chloride of ammonium, and ammonia* precipitates only concentrated solutions.

Acetate of lead throws down white phosphite of lead, insoluble in acetic acid.

By boiling with *sulphurous acid* in excess, phosphoric acid is formed with separation of sulphur.

In contact with *zinc and dilute sulphuric acid* phosphorous acid gives a mixture of hydrogen and phosphuretted hydrogen, which fumes in the air, burns with an emerald color, and precipitates phosphide of silver from solution of nitrate of silver.

Hypophosphorous Acid.

The solution of the acid is resolved by boiling with exclusion of air, into phosphoric acid and phosphuretted hydrogen gas, which is not spontaneously inflammable. All hypophosphites are resolved by ignition into phosphate and phosphuretted hydrogen gas, which in most cases is spontaneously inflammable.

Chloride of barium, chloride of calcium, and *acetate of lead* fail to precipitate hypophosphites. (Here they differ from phosphites.)

Nitrate of silver gives with hypophosphites, first, a white precipitate of hypophosphite of silver, which turns black even at the common temperature, but more rapidly on heating; the change of color being attended with separation of metallic silver.

Mercuric chloride added in excess to hypophosphorous acid is reduced to mercurous chloride. The change is favoured by heat.

With *zinc and sulphuric acid* hypophosphorous acid gives hydrogen gas mixed with phosphuretted hydrogen.

Sulphurous Acid.

When treated with *hydrochloric acid* all sulphites evolve sulphurous anhydride, which may be known by its odor.

Chlorine water converts sulphurous acid to sulphuric acid.

Chloride of barium precipitates neutral sulphites; the precipitate dissolves in hydrochloric acid.

Hydrosulphuric acid decomposes sulphurous acid, water and pentathionic acid being formed, and free sulphur eliminated.

If to a solution of sulphurous acid mixed with an equal volume of hydrochloric acid, a piece of clean *copper* wire is added, and the mixture is boiled, the copper appears black, as if covered with soot, if much sulphurous acid is present; but only dull if a little is present.

If a trace of a sulphite is introduced into a *hydrogen apparatus,* hydrosulphuric acid is immediately evolved with the hydrogen, and the gas now produces a black precipitate in a solution of acetate of lead, to which has been added sufficient potash to redissolve the precipitate first formed.

Sulphurous acid is a powerful reducing agent; it reduces *chromic acid, permanganic acid,* and *mercuric chloride,* and it decolorizes *iodide of starch.*

With a hydrochloric acid solution of *stannous chloride* a yellow precipitate of stannic sulphide is formed after some time.

If an aqueous solution of an alkaline sulphite is mixed with acetic acid just to give it an incipient acid reaction, and is then added to a relatively large amount of solution of *sulphate of zinc,* mixed with a very small quantity of *nitroprusside of sodium,* the fluid acquires a red color if the quantity of sulphite present is not too inconsiderable; but when the quantity of sulphite is very minute, the coloration does not make its appearance till some ferrocyanide of potassium has been added. The latter salt occasions the formation of a purple-red precipitate, unless the quantity of sulphite is too slight. (Hyposulphites of the alkali metals do not show this reaction.)

Hyposulphurous Acid.

The solutions of most hyposulphites may be boiled without suffering decomposition. Hyposulphite of calcium is resolved upon boiling into sulphite of calcium and sulphur.

If *hydrochloric acid* is added to the solution of a hyposulphite, the fluid remains at first clear and odorless, but after a short time—the shorter the more concentrated the solution—it becomes more and more turbid, owing to the separation of sulphur, and exhales the odor of sulphurous acid. Application of heat promotes the decomposition.

Nitrate of silver produces a white precipitate of hyposulphite of silver, which is soluble in an excess of the hyposulphite; after a little while (upon heating almost immediately) this precipitate turns black, being decomposed into sulphide of silver and sulphuric acid. Hyposulphite of sodium dissolves chloride of silver; upon addition of an acid the solution remains clear at first, but after some time, and immediately upon boiling, sulphide of silver separates.

Chloride of barium produces a white precipitate, which is soluble in much water, more especially hot water, and is decomposed by hydrochloric acid.

Ferric chloride colors the solutions of alkaline hyposulphites reddish-violet (here they differ from alkaline sulphites); on standing, the liquid is decolorized, especially when heated, ferrous chloride being produced.

Hyposulphites immediately reduce an acidified solution of *chromic acid*, and decolorize acidified *iodide of starch.*

WHERE IT IS REQUIRED TO FIND SULPHITES AND HYPOSULPHITES OF THE ALKALI METALS IN PRESENCE OF ALKALINE SULPHIDES, solution of sulphate of zinc first is added, until the sulphide is decomposed; the sulphide of zinc is then filtered off, and one part of the filtrate is tested for hyposulphurous acid with hydrochloric acid, while another portion is tested for sulphurous acid with nitroprusside of sodium, &c.

REACTIONS AND SEPARATION

OF

ORGANIC BODIES.

REACTIONS OF ALKALOIDS.

The alkaloids may be classified as follows :—

VOLATILE ALKALOIDS.

Obtained in the distillate, when their salts are distilled with potash and water.

Nicotine.
Conine.

NON-VOLATILE ALKALOIDS.

Group I.

Precipitated by potash from their salts, readily soluble in excess of the precipitant.

Morphine.

Group II.

Precipitated by potash from their salts, insoluble in excess of the precipitant.

Precipitated by acid carbonate of sodium, even from acid solution, if the solution does not contain less than 1 *per cent.*

Narcotine.
Quinine.
Cinchonine.

Group III.

Precipitated by potash from their salts, insoluble in excess of the precipitant.

Not precipitated by acid carbonate of sodium from acid solution.

Strychnine.
Brucine.
Veratrine.

VOLATILE ALKALOIDS.

NICOTINE. CONINE.

Nicotine.

1. Nicotine is a colorless, oily liquid, of 1·048 sp. gr.; it becomes yellow or brown by the action of air. It boils at 250°, being partially decomposed; but in a current of hydrogen it distils over unchanged between 100° and 200°. It is perfectly miscible with water, alcohol, and ether.

Nicotine has a peculiar, somewhat ethereal, tobacco-like odor (the odor is much increased by heating), and an acrid, pungent taste. It is very poisonous. Dropped on paper, it makes a transparent stain, which slowly disappears; it is alkaline to test-paper. A strong aqueous solution of the alkaloid shows these reactions more distinctly than pure nicotine.

2. The salts of nicotine are freely soluble in water and alcohol, insoluble in ether; they are inodorous, but taste strongly of tobacco; some of them are crystallizable. By distilling the salts with potash, neutralizing the distillate with oxalic acid, and evaporating, oxalate of nicotine is left, which may be freed from any oxalate of ammonium that may be present by means of alcohol, in which it is soluble, oxalate of ammonium being insoluble.

3. If a solution of a nicotine salt is mixed with *potash* and then shaken with *ether*, the nicotine will be dissolved by the ether. On allowing the ethereal solution to evaporate on a watch-glass, the nicotine will be left, and on warming the watch-glass, the alkaloid will volatilize in white, strong-smelling fumes.

4. *Chloride of platinum* produces in solutions of nicotine a whitish-yellow, flocculent precipitate. On heating the mixture, the precipitate dissolves, but on continuing the application of heat, the precipitate soon forms again as an orange-yellow, crystalline, heavy powder, which, under the microscope, appears to be composed of rounded crystalline grains. If a rather dilute solution of nicotine, supersaturated with hydrochloric acid, is mixed with chloride of platinum, the fluid at first remains clear; after some time, however, the double salt sepa-

rates in small crystals (oblique, four-sided prisms), clearly discernible with the naked eye.

5. *Chloride of gold* produces a reddish-yellow, flocculent precipitate, sparingly soluble in hydrochloric acid.

6. Solution of *iodine in iodide of potassium,* added in small quantity to solution of nicotine, produces a yellow precipitate, which after a time disappears. Upon further addition of the reagent, a copious, kermes-colored precipitate separates; but this also disappears after a time.

7. *Tannic acid* produces a copious white precipitate, which redissolves upon addition of hydrochloric acid.

8. If a solution of nicotine is added to *mercuric chloride* in excess, a white, flocculent precipitate is formed. On adding chloride of ammonium, the precipitate redissolves. But the fluid soon turns turbid, and deposits a heavy, white precipitate.

Conine.

1. Conine is a colorless oily liquid, of ·87 sp. gr.; it becomes brown by the action of air. It boils at 212°; when distilled in the ordinary way it turns brown, and is partially decomposed; heated in a current of hydrogen, it distils unchanged; with aqueous vapours it distils freely. It dissolves but sparingly in water, 1 part requiring 100 parts of water at the common temperature; the solution becomes turbid on warming, and clear again on cooling. Conine is perfectly miscible with alcohol and ether. The aqueous and alcoholic solutions are strongly alkaline to test paper. Conine has a very strong, pungent, repulsive odor, which affects the head, and an unpleasant taste. It is very poisonous.

2. The salts of conine are soluble in water and alcohol; some are to a certain extent soluble in ether. Hydrochlorate of conine crystallizes readily; the smallest quantity of conine, brought in contact with a trace of hydrochloric acid, yields almost immediately non-deliquescent rhombic crystals. The sulphate of conine does not crystallize till after some time. The solutions of conine salts turn brownish on evaporation, with partial decomposition of the alkaloid. The dry salts do not smell of conine; when moistened they smell but feebly of it; but upon addition of potash they at once give the characteristic smell. By distilling the salts with potash, neutralizing

the distillate with oxalic acid, evaporating to dryness, and treating the residue with spirit of wine, the oxalate of conine is dissolved, whilst any oxalate of ammonium that may be present is left undissolved. As conine is only sparingly soluble in water, and still less soluble in alkaline solutions, a strong solution of a conine salt turns milky on addition of potash. The minute drops which separate unite gradually, and collect on the surface.

3. If a solution of a conine salt is shaken with *potash and ether*, the conine dissolves in the ether. If the latter is then allowed to evaporate on a watch-glass, the conine is left in yellowish oily drops.

4. *Strong nitric acid* gives to conine a fine blood-red color.

5. *Sulphuric acid* gives a purple-red color, which subsequently turns olive-green.

6. *Chloride of platinum* does not precipitate salts of conine. The conine compound corresponding to chloride of platinum and ammonium is soluble in water, but insoluble in alcohol and in ether. The double salt dissolves on boiling with alcohol, and separates on cooling in the amorphous form.

7. *Chloride of gold* produces a yellowish-white precipitate, insoluble in hydrochloric acid.

8. *Mercuric chloride* produces a copious white precipitate, soluble in hydrochloric acid.

9. *Iodine dissolved in iodide of potassium*, and also *tannic acid* give the same reactions as with nicotine.

10. *Chlorine* water produces in a mixture of water and conine, a strong white turbidity.

11. If a solution of conine is mixed with solution of *albumen* the latter coagulates. Aniline is the only other volatile alkaloid which shows this reaction.

NON-VOLATILE ALKALOIDS.

GROUP I.

Morphine.

1. Crystallized morphine usually appears in the form of colorless, brilliant, rhombic prisms. It has a bitter taste, and dissolves very sparingly in cold, but somewhat more readily in boiling water. It dissolves in about 90 parts of cold

alcohol, and from 20 to 30 parts of boiling alcohol. The solutions both in alcohol and in hot water have a distinctly alkaline reaction. Morphine is nearly insoluble in ether; it dissolves in amyl alcohol in the cold, but more freely on heating. It is insoluble in benzol, very difficultly soluble in chloroform. Morphine may be sublimed without decomposition.*

2. The salts of morphine are readily soluble in water and alcohol, but insoluble in ether and amyl alcohol. Their taste is disagreeably bitter. Most of them are crystallizable.

3. *Potash* and *ammonia* precipitate the alkaloid from solutions of its salts, in the form of a white crystalline powder. The formation of the precipitate generally takes some time; it is accelerated by stirring and rubbing. The precipitate redissolves with great readiness in excess of potash, but more sparingly in ammonia. It dissolves also in chloride of ammonium, and in carbonate of ammonium, but with difficulty in the latter.

4. *Carbonate of potassium*, or of sodium, produces the same precipitate, which is insoluble in excess of either reagent. Consequently, if acid carbonate of potassium or of sodium is added to, or if carbonic acid is passed into, a solution of morphine in potash, the morphine separates, especially after boiling, in the form of a crystalline powder. This powder slightly magnified appears as acicular crystals, and magnified one hundred times as rhombic prisms.

5. *Acid carbonate of potassium*, or of sodium, speedily produces in neutral solutions a precipitate of morphine, insoluble in excess of the reagent. Acidified solutions of morphine are not precipitated in the cold.

6. *Strong nitric acid* added to salts in the solid state or in strong solutions, produces a color varying from red to yellowish-red. Dilute solutions do not change color on addition of nitric acid in the cold, but become yellow upon heating.

7. If morphine, or a salt of morphine, is treated with 4 or

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* For the best way of subliming morphine, and for the value of the sublimate in microscopic diagnosis, see Helwig (Zeitschr. f. anal. Chem., 3, 43; or Das Mikroscop in der Toxikologie von Dr. A. Helwig. von Zabern. Mainz: 1864). In the latter work the subject is treated more completely and illustrated. I may mention that the alkaloid must be perfectly pure.

5 drops of pure strong sulphuric acid, and warmed, a colorless solution is obtained; if, after cooling, 10 to 20 drops of sulphuric acid, mixed with nitric acid,* are added, and 2 or 3 drops of water, the fluid acquires a violet-red color (gentle heating promotes the reaction); and if now 4 or 5 clean lentil-sized fragments of binoxide of manganese are added, or a fragment of chromate of potassium (Otto), the fluid acquires an intense mahogany color. If the fluid is then diluted with 4 parts of water, cooled in a test-tube, and ammonia added till the reaction is almost neutral, a dirty yellow color makes its appearance, which turns brownish red upon supersaturation with ammonia, without the deposition of any appreciable precipitate.

According to A. Husemann the violet coloration of sulphate of morphine by nitric acid, does not occur till the morphine solution has undergone change. It occurs immediately when the solution in strong sulphuric acid is heated to 100°—150°. If, after cooling, a drop of nitric acid is added, a splendid dark violet color is produced, which stays at the edge for several minutes, but in the middle soon passes into a blood-red color, which slowly becomes paler. Chloride of soda acts like nitric acid. (Very delicate reaction.) On heating morphine with sulphuric acid above 150°, a transient reddish violet color is produced, which turns finally to dirty green.

8. Neutral *ferric chloride* gives to strong neutral solutions of morphine a beautiful dark blue color, which disappears on the addition of an acid. If the solution contains animal or vegetable extractive matters or acetates, the color will appear clouded and less distinct.

9. *Iodic acid* added to morphine or a morphine salt, is decomposed, with separation of iodine. Other nitrogenous bodies (albumen, casein, fibrin, &c.) reduce iodic acid in the same manner. In the case of morphine, the coloration is made more intense by the addition of ammonia; in the case of other bodies, the color is removed by the addition of ammonia.†

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* Mix 3 drops of strong nitric acid with 100 c.c. water, and add 10 drops of this to 40 c.c. of pure strong sulphuric acid.

† For the detection of small quantities of morphine, Lefort recommends to moisten strips of white blotting-paper repeatedly with the solution, drying after each moistening. Nitric acid, ferric chloride, and iodic acid and ammonia, will give the reactions on the paper plainly.

GROUP II.

NARCOTINE. QUININE. CINCHONINE.

Narcotine.

1. Narcotine appears usually as colorless brilliant rhombic prisms, or a white loose crystalline powder. It is insoluble in water. Alcohol and ether dissolve it sparingly in the cold, but somewhat more readily on heating. The solid narcotine is tasteless, the alcoholic and ethereal solutions are intensely bitter. The alkaloid does not alter vegetable colors. It fuses at 170°.

2. The salts of narcotine have invariably an acid reaction. Those with weak acids are decomposed by a large amount of water, and, if the acid is volatile, even by simple evaporation. Most of the salts are amorphous and soluble in water, alcohol, and ether; they have a bitter taste.

3. *The alkalies, their carbonates and their acid carbonates* immediately precipitate narcotine from solutions of its salts, in the form of a white powder, which when magnified one hundred times, appears as an aggregate of crystalline needles. The precipitate is insoluble in excess of either precipitant. If a solution of a narcotine salt is mixed with ammonia, and shaken with ether, the alkaloid dissolves in the latter. If a drop of the ethereal solution is evaporated on a watch-glass, the residue is seen, when magnified one hundred times, to consist of distinct needles.

4. *Strong nitric acid* dissolves narcotine to a colorless fluid, which becomes yellow on heating.

5. *Strong sulphuric acid* acts differently upon different specimens of narcotine. Those that are apparently the purest give a bluish violet solution which in a short time becomes dirty orange; specimens which appear less pure give a yellow solution at once. If the yellow solution in either case is warmed very gradually it becomes at first orange red, subsequently beautiful bluish stripes are produced, and finally, when the sulphuric acid begins to evaporate, an intense reddish violet color is formed. If the heating is interrupted when the blue color is present, the solution slowly becomes cherry red in the cold. The reaction is very delicate. (Husemann.*)

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* Zeitschr. f. anal. Chem., 3, 151.

6. If to a solution of narcotine in strong sulphuric acid prepared in the cold, 10 to 20 drops of sulphuric acid containing a minute quantity of nitric acid (footnote, p. 204) are added, and then two or three drops of water, the fluid becomes intensely red. Addition of binoxide of manganese does not change the color. If the solution is diluted and ammonia is added in excess, a dark brown precipitate is produced. If to a solution of narcotine in strong sulphuric acid, prepared in the cold, chloride of soda is added, a distinct and rather permanent crimson color is produced, which passes into yellowish red. The solution of narcotine in strong sulphuric acid, which has been colored by heat, is turned immediately bright yellow by nitric acid or chloride of soda, and a more reddish coloration appears gradually (Husemann).

7. *Chlorine-water* added to solution of a narcotine salt gives a yellow color, slightly inclining to green. On the addition of ammonia a yellowish red and much more intense color is produced.

8. If a salt of narcotine is dissolved in excess of dilute sulphuric acid, mixed with finely powdered binoxide of manganese, and boiled for some minutes, the alkaloid is decomposed. On filtering, and adding ammonia to the filtrate, no precipitate will be obtained.

Quinine.

1. Quinine appears either in the form of fine crystalline needles of silky lustre, which are frequently aggregated into tufts, or as a loose powder. It is sparingly soluble in cold, but somewhat more readily in hot water. It is readily soluble in alcohol, both cold and hot, but less soluble in ether. The taste of quinine is intensely bitter. The solutions manifest alkaline reaction. Upon exposure to heat it loses water.

2. The salts of quinine are intensely bitter. Most of them are crystallizable, difficultly soluble in cold water, more readily soluble in hot water and alcohol. The acid salts dissolve very freely in water; the solutions reflect a bluish tint.

3. *The alkalies and their carbonates* produce, in solutions of the salts (if not too dilute), a white loose pulverulent precipitate of quinine, which immediately after precipitation ap-

pears opaque and amorphous under the microscope, but assumes, after the lapse of some time, the appearance of an aggregate of crystalline needles. The precipitate redissolves only to a scarcely perceptible extent in excess of potash, but it is more soluble in ammonia. It is hardly more soluble in potassium or sodium carbonate than in water. If a solution of a quinine salt is mixed with ammonia, and shaken with ether, the latter dissolves the quinine. In this last reaction quinine differs essentially from cinchonine.

4. *Acid carbonate of sodium* produces in neutral and acid solutions of quinine a white precipitate. If the solution contains 1 per cent. of the alkaloid, a precipitate appears immediately; if the solution contains ·7 per cent., the precipitate separates only after an hour or two; and if the solution contains ·5 per cent., the fluid remains clear at first, and a slight precipitate forms in the course of twelve or twenty-four hours. The precipitate is not altogether insoluble in excess of the precipitant; it is well, therefore, not to have much excess. The precipitate contains carbonic acid.

5. *Strong nitric acid* dissolves quinine to a colorless fluid, turning yellowish upon application of heat.

6. *Chlorine water* produces no apparent change until ammonia is added, when a deep emerald green color makes its appearance. If, after the addition of chlorine water, some ferrocyanide of potassium is added, and then a few drops of ammonia, the fluid acquires a magnificent deep red tint, which soon turns to dirty brown. This reaction is delicate and characteristic. Upon addition of an acid to the red fluid, the color vanishes, but reappears afterwards upon cautious addition of ammonia.

7. *Strong sulphuric acid* dissolves the pure salts of quinine to a colorless or very faint yellowish fluid, which turns yellow or brown on the application of heat. Sulphuric acid, containing some nitric acid, dissolves quinine to a colorless or very faint yellowish fluid.

8. As regards the polarizing properties of iodide of sulphate of quinine, *see* "Phil. Mag.," 6, 171.

Cinchonine.

1. Cinchonine appears either as transparent brilliant

rhombic prisms, or fine white needles, or a loose powder. At first it is tasteless, but after some time the bitter taste becomes perceptible. It is nearly insoluble in cold water, and dissolves only with extreme difficulty in hot water; it dissolves sparingly in cold dilute alcohol, more readily in hot alcohol, and freely in absolute alcohol. From hot alcoholic solutions the greater portion of the alkaloid separates upon cooling in a crystalline form. Solutions of cinchonine taste bitter, and manifest alkaline reaction. Cinchonine is insoluble in ether.* Cinchonine may be sublimed, preferably in a current of hydrogen.

2. The salts of cinchonine have a bitter taste; most of them are crystallizable; they are generally more readily soluble in water and alcohol than the corresponding quinine salts. They are insoluble in ether.

3. *The alkalies and their carbonates* produce a white loose precipitate of cinchonine, which does not redissolve in excess of any of the precipitants. If the solution was concentrated, the precipitate does not appear distinctly crystalline—even when magnified 200 times; if the solution was very dilute, and the precipitate only formed after some time, it appears under the microscope to consist of distinct needles aggregated into tufts.

4. *Acid carbonate of potassium* or of sodium precipitates cinchonine both from neutral and acidified solutions of its salts, but not so completely as the carbonate of potassium or sodium. A precipitate forms immediately in solutions containing only ·5 per cent. of cinchonine

5. *Strong sulphuric acid* dissolves cinchonine to a colorless fluid, which on heating turns black. Addition of nitric acid leaves the solution colorless in the cold, but on heating a black color is developed.

6. *Chlorine water* gives no color to a solution of a cinchonine salt. If ammonia is also added, a yellowish white precipitate is formed.

7. If a solution of a cinchonine salt containing very little or no free acid, is mixed with *ferrocyanide of potassium*, a

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* The cinchonine of commerce usually contains another alkaloid, called cinchotine, which is soluble in ether. Cinchotine crystallizes in large rhomboidal crystals of brilliant lustre, which fuse at a high temperature, and cannot be sublimed, even in a current of hydrogen.

flocculent precipitate of hydroferrocyanide of cinchonine is formed. If an excess of the precipitant is added, and a gentle heat very slowly applied, the precipitate dissolves, but separates again upon cooling, in brilliant golden scales, or in long needles, often aggregated in the shape of a fan. With the aid of a microscope this reaction is as delicate as it is characteristic.

Group III.

STRYCHNINE. BRUCINE. VERATRINE.

Strychnine.

1. Strychnine appears in the form of brilliant rhombic prisms, or a white powder. It has an extremely bitter taste. It is nearly insoluble in cold, and barely soluble in hot water. It is almost insoluble in absolute alcohol and ether, and only sparingly soluble in dilute alcohol. It dissolves freely in amyl alcohol, especially with the aid of heat. It is also soluble in benzol and chloroform. By cautious heating it may be sublimed unchanged (Helwig, see note, p. 203).

2. The salts of strychnine are for the most part crystallizable; they are soluble in water. All the salts have an intolerably bitter taste.

3. *Potash* produces in solutions of strychnine salts a precipitate of strychnine, which is insoluble in excess of potash. Magnified 100 times it is seen to consist of needles. From dilute solutions the precipitate separates only after some time, and then has the form of needles visible to the naked eye.

4. *Carbonate of sodium* produces the same precipitate, insoluble in excess.

5. *Ammonia* produces the same precipitate. It redissolves in excess of ammonia; but after some time the alkaloid separates in the form of needles, visible to the naked eye.

6. *Acid carbonate of sodium* produces in neutral solutions a precipitate of strychnine, which separates in fine needles shortly after the addition of the reagent, and is insoluble in an excess of the precipitant. But upon adding one drop of acid (so as to leave the fluid still alkaline), the precipitate dissolves readily in the liberated carbonic acid. The addition

of acid carbonate of sodium to an acid solution of strychnine causes no precipitation; and it is only after the lapse of twenty-four hours, or more, that the alkaloid crystallizes from the fluid in distinct prisms, in proportion as the free carbonic acid escapes. If a concentrated solution of strychnine, supersaturated with acid carbonate of sodium, is boiled for some time, a precipitate forms at once; from dilute solutions this precipitate separates only after concentration.

7. *Sulphocyanide of potassium* produces in concentrated solutions of salts of strychnine immediately, in dilute solutions after some time, a white crystalline precipitate, which appears under the microscope as an aggregate of flat needles, truncated or pointed at an acute angle, and is but little soluble in an excess of the precipitant.

8. *Mercuric chloride* produces in solutions of salts of strychnine a white precipitate, which changes after some time to crystalline needles, aggregated into stars, and distinctly visible through a lens. Upon heating the fluid these crystals redissolve, and upon subsequent cooling the double compound recrystallizes in larger needles.

9. If a few drops of *strong sulphuric acid* are added to a little strychnine in a porcelain dish, solution ensues, without coloration of the fluid. If small quantities of oxidizing agents (chromate of potassium, permanganate of potassium, ferricyanide of potassium, peroxide of lead, binoxide of manganese) are now added, in the solid form, the fluid acquires a magnificent bluish-violet color, which, after some time, changes to wine-red, then to reddish-yellow. With chromate of potassium and permanganate of potassium the reaction is immediate; on inclining the dish, blue violet streaks are seen to flow from the salt fragment, and by pushing the latter about the coloration is soon imparted to the entire fluid. With ferricyanide of potassium the reaction is less rapid; but it is slowest with peroxides. The more speedy the manifestation of the reaction the more rapid is also the change of color from one tint to another. I prefer chromate of potassium or permanganate of potassium to all other oxidizing agents. Jordan succeeded, with chromate of potassium, in distinctly showing the presence of $\frac{1}{50000}$th grain of strychnine. J. Erdmann prefers binoxide of manganese in lentil-sized fragments. Metallic chlorides and considerable quantities of nitrates, also

large quantities of organic substances, prevent the manifestation of the reaction or impair its delicacy. It is therefore always advisable to free the strychnine first, as far as practicable, from all foreign matters before proceeding to try this reaction.

Morphine interferes with this reaction.* In order to remove the morphine, the concentrated aqueous neutral solution of the substance is mixed with ferricyanide of potassium or neutral chromate of potassium and filtered. The precipitate contains the strychnine, the solution the morphine. The precipitate is washed a little, dried, and mixed in a watch-glass with strong sulphuric acid. The blue-violet color is immediately produced. It should be borne in mind that the strychnine precipitate is not insoluble in water.†

10. Strong *chlorine water* produces in solutions of strychnine salts a white precipitate, which is soluble in ammonia to a colorless fluid.

11. *Strong nitric acid* dissolves strychnine and its salts to a colorless fluid, which turns yellow when heated.

Brucine.

1. Brucine appears either in the form of transparent straight rhombic prisms, or in that of crystalline needles aggregated into stars, or as a white powder composed of minute crystalline scales. Brucine is difficultly soluble in cold water, but somewhat more readily in hot. It dissolves freely in alcohol, both absolute and dilute; it dissolves freely in cold, but more readily in hot amyl alcohol; but it is almost insoluble in ether. Its taste is intensely bitter. It may be sublimed. (See Note p. 203.)

2. The salts of brucine are readily soluble in water, and of an intensely bitter taste. Most of them are crystallizable.

3. *Potash* throws down brucine from its salts; the precipitate is insoluble in excess of the precipitant. Viewed

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* See Reese, Zeitschr. f. anal. Chem., 1, 399. Horsley, *Ibid.* 1, 515. Thomas, *Ibid.* 1, 517.

† Rodgers recommends to separate strychnine from morphine by benzol, in which the former alone is soluble. Thomas recommends to render the solution of the acetates alkaline with potash, and to shake with chloroform; the morphine remains in the alkaline solution, while the strychnine dissolves in the chloroform.

under the microscope, immediately after precipitation, it appears to consist of very minute grains; but upon further inspection, these grains are seen—with absorption of water—to suddenly form into needles, which latter subsequently arrange themselves without exception into concentric groups. These successive changes of the precipitate may be distinctly traced even with the naked eye.

4. *Carbonate of sodium* produces the same effect as potash.

5. *Ammonia* produces in solutions of brucine salts a whitish precipitate, which appears at first like a number of minute drops of oil, but changes subsequently, with absorption of water, to small needles. The precipitate redissolves, immediately after separation, very readily in an excess of the precipitant; but after a very short time, unless the solution is very dilute, the brucine crystallizes out in small concentrically grouped needles, which are insoluble in excess of ammonia.

6. *Acid carbonate of sodium* precipitates brucine from neutral solutions of its salts. The precipitate separates after some time in the form of concentrically aggregated needles of silky lustre, which are insoluble in an excess of the precipitant, but dissolve in free carbonic acid (compare strychnine). Acid carbonate of sodium fails to precipitate acid solutions of brucine salts; and it is only after a long time, and with escape of carbonic acid, that the alkaloid separates in regular and comparatively large crystals.

7. *Strong nitric acid* dissolves brucine and its salts to intensely red fluids, which subsequently acquire a yellowish-red color, and turn yellow when heated. Upon addition of stannous chloride or sulphide of ammonium to the heated fluid, no matter whether it remains concentrated or has been diluted, the faint yellow color turns to a most intense violet.

8. If a little brucine is treated with 4 or 5 drops of *strong sulphuric acid,* a solution of a faint rose color is obtained, which afterwards turns yellow. If 10 or 20 drops of sulphuric acid mixed with nitric acid (see Note p. 204) are now added, the fluid transiently acquires a red, afterwards a yellow tint. Addition of binoxide of manganese transiently imparts a red, then a gamboge tint to the fluid. If the fluid is then, with proper cooling, diluted with 4 parts of water and mixed with ammonia to alkaline reaction, a golden-yellow color is formed (J. Erdmann).

9. *Chlorine water* gives a bright red color to solutions of brucine salts. If ammonia is then added, the color changes to yellowish brown.

10. *Sulphocyanide of potassium* produces in strong solutions immediately, in dilute solutions after some time, a granular crystalline precipitate, which, under the microscope, appears composed of variously aggregated polyhedral crystalline grains. Friction promotes the separation of the precipitate.

11. *Mercuric chloride* produces a white granular precipitate, which, under the microscope, appears composed of small roundish crystalline grains.

Veratrine.

1. Veratrine appears in the form of small prismatic crystals, which acquire a porcelain-like appearance in the air, or in the form of a white powder, of acrid and burning, but not bitter taste. It is exceedingly poisonous. The most minute quantity of veratrine is capable of exciting most violent sneezing. It is insoluble in water. In alcohol it dissolves freely; but in ether it is not so soluble. It fuses like wax at 115°, and solidifies on cooling to a transparent yellow mass. By cautious heating it may be sublimed unchanged (see Note, p. 203).

2. Some of the salts of veratrine are crystallizable, others dry up to a gummy mass. They are soluble in water, and have an acrid burning taste.

3. *Potash, ammonia,* and the *alkaline carbonates* produce in solutions of veratrine salts a flocculent white precipitate, which, immediately after precipitation, does not appear crystalline under the microscope. After a few minutes, however, it alters its appearance, and small scattered clusters of short prismatic crystals are observed, instead of the original flakes. The precipitate does not redissolve in excess of potash or carbonate of potassium. It is slightly soluble in ammonia in the cold, but the dissolved portion separates again upon application of heat.

4. *Acid carbonate of sodium* produces the same effect with veratrine salts as with those of strychnine and brucine. However, the veratrine separates readily upon boiling, even from dilute solutions.

5. *Strong nitric acid* causes veratrine to agglutinate into small resinous lumps, which afterwards dissolve slowly in the acid. If the alkaloid is pure, the solution is colorless.

6. *Strong sulphuric acid* also causes veratrine to agglutinate into lumps; but these dissolve with great readiness to a faint yellow fluid, the color of which gradually increases in depth, and changes afterwards to a reddish yellow, and then to an intense blood-red. The color lasts two or three hours, then gradually disappears. Addition of sulphuric acid containing nitric acid, or of binoxide of manganese causes no great change of color. If the fluid is then diluted with water, and ammonia added until the reaction is nearly neutral, a yellowish solution is obtained, in which ammonia added in excess produces a light-brown precipitate. (J. Erdmann.)

7. If veratrine is dissolved in *strong hydrochloric acid,* a colorless fluid is obtained, which by long boiling acquires an intensely red tint, permanent on standing. The reaction is very delicate, and occurs not only with the perfectly pure veratrine but with the ordinary commercial alkaloid. (Trapp.)

8. *Sulphocyanide of potassium* produces only in concentrated solutions a flocculent-gelatinous precipitate.

9. *Chlorine water* added to solutions of salts of veratrine, gives a yellowish color, which turns to a faint brown on addition of ammonia. In concentrated solutions, chlorine produces a white precipitate.

Although *salicine* is not an alkaloid, its reactions may find a place here.

Salicine.

1. Salicine appears either in the form of white needles and scales of silky lustre, or, where the crystals are very small, as a powder of silky lustre. It has a bitter taste, is readily soluble in water and in alcohol, but is insoluble in ether.

2. No reagent precipitates salicine as such.

3. If salicine is treated with *strong sulphuric acid,* it agglutinates into a resinous lump, and acquires an intense blood-red color, without dissolving in the acid; the color of the acid is at first unaltered.

4. If an aqueous solution of salicine is mixed with *hydro-*

chloric acid or *dilute sulphuric acid* and boiled for a short time, the fluid suddenly becomes turbid, and deposits a fine granular crystalline precipitate (saliretine). If to the mixed fluid and precipitate a drop or two of chromate of potassium is added, and the whole is boiled, the saliretine acquires a fine red color, while the fluid turns green from reduction of the chromic acid. (Bouxlier.)

SEPARATION OF THE ALKALOIDS.

THE VOLATILE ALKALOIDS.

The volatile alkaloids are most easily recognised when isolated. They are isolated by distilling with potash, neutralizing the distillate with oxalic acid, evaporating, taking up the residue with alcohol, evaporating, treating with water, shaking with potash and ether, pouring off the ethereal solution, and allowing it to evaporate spontaneously.

The two alkaloids are distinguished from each other chiefly by their odor, their solubility in water, and their behaviour with chlorine water, with chloride of platinum, and with albumen.

THE NON-VOLATILE ALKALOIDS.

Group II.

To separate the three members of this group from each other, the aqueous solution is mixed with excess of ammonia, and shaken with ether; the ethereal solution is then poured off. The cinchonine is left in the aqueous fluid; the narcotine and quinine dissolve in the ether. The ethereal solution is evaporated, the residue dissolved in hydrochloric acid, diluted to contain ·5 per cent. of the alkaloids, and mixed with acid carbonate of sodium. The narcotine is precipitated, while the quinine remains in solution. By filtering, evaporating the solution, and treating the residue with water, the quinine is left behind in the pure state.*

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* If it is wanted to separate quinine not only from cinchonine, but also from the other bark alkaloids (α quinidine, β quinidine, γ quinidine and cinchonidine), this process with ammonia and ether is inadequate, since some of these other alkaloids are pretty freely soluble in ether (Kerner, Zeitschr. f. anal. Chem., 1, 150). Kerner gives a process based on the fact that the quinine thrown down by ammonia from a solution of the sulphate requires less ammonia to redissolve it than all the other bark alkaloids. For the separation of quinine from quinidine see also Schwarzer (Op. cit., 4, 129); and for the separation of the bark alkaloids in general see van der Burg (Op. cit., 4, 273).

Group III.

Strychnine may be separated from brucine and veratrine by absolute alcohol, in which it is insoluble, and brucine and veratrine are readily soluble. Brucine and veratrine are not easily separated from each other, but may be detected in the presence of each other.

The identity of strychnine is best established by the reaction with sulphuric acid and an oxidizing agent,* by the form of its crystals when thrown down by alkalies, and seen under the microscope, and by the form of the precipitates with sulphocyanide of potassium and mercuric chloride.

The identity of brucine is best established by the reactions with nitric acid and stannous chloride or sulphide of ammonium, or by the form of the crystalline precipitate produced by ammonia.

Veratrine is sufficiently distinguished from brucine and from all the other alkaloids mentioned, by its characteristic deportment at a gentle heat, and also by the form of the precipitate produced by alkalies. To distinguish veratrine in presence of brucine, the reaction with strong sulphuric acid or with hydrochloric acid is employed.

Detection of One of the Five Poisonous Alkaloids (Morphine, Narcotine, Strychnine, Brucine, Veratrine), when Present in the Pure State, and in the Solid Form. (J. Erdmann.)

1. Treat the substance with 4 or 5 drops of pure strong sulphuric acid.

   A yellow color, soon changing to red. Veratrine.

   A rose color, changing gradually to yellow. Brucine.

   (Morphine, narcotine,† and strychnine if pure, give no color to sulphuric acid).

2. No matter whether there is color or not, add to the fluid

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* Aniline shows somewhat similar reactions when treated with sulphuric acid and oxidizing agents. It at first gives a pale green color, which turns gradually to a magnificent blue, and finally, after some time, black.

† Husemann says that many narcotines do color sulphuric acid. See p. 205, 5.

obtained in 1, from 10 to 20 drops of strong sulphuric acid containing nitric acid (see note, p. 204), then 2 or 3 drops of water. After twenty or thirty minutes, the fluid shows

A violet-red color. MORPHINE.

An onion-red color. NARCOTINE.

A yellow color (after a transient red). BRUCINE.

With VERATRINE the red color of the sulphuric acid solution is not materially altered.

With STRYCHNINE no coloration is observed.

3. Put into the fluid obtained in 2, whether colored or not, 4 or 5 clean fragments of binoxide of manganese, of the size of a lentil. After an hour the fluid shows

A mahogany color. MORPHINE.

A yellowish-red to blood-red color. NARCOTINE.

A deep onion-red color (after a transient purple violet). STRYCHNINE.

A gamboge color (after a transient red). BRUCINE.

A dark cherry-red color. VERATRINE.

4. Pour the colored fluid obtained in 3, into a test tube containing four times the volume of water, and add ammonia till the acid is almost neutralized. (Heat must be avoided as much as possible in these operations.)

A dirty yellow color, changing to brownish-red upon supersaturation with ammonia, without immediate deposition of a notable precipitate. MORPHINE.

A reddish coloration; upon supersaturation with ammonia, a copious dark-brown precipitate. NARCOTINE.

A violet purple color, turning yellowish-green to yellow on supersaturation with ammonia. STRYCHNINE.

A golden yellow color, not materially changed by excess of ammonia. BRUCINE.

A faint brownish color, turning yellowish upon supersaturation with ammonia, a light brown precipitate being deposited. VERATRINE.

DETECTION OF ONE OF THE NON-VOLATILE ALKALOIDS, OR OF SALICINE, WHEN PRESENT IN AN AQUEOUS SOLUTION ALONE.

1. Add dilute potash, drop by drop, to a portion of the

solution, until the fluid acquires a scarcely perceptible alkaline reaction; stir, and allow to stand some time.

No precipitate is formed. Absence of alkaloids, probable presence of SALICINE. Test the original substance with strong sulphuric acid, and also with hydrochloric acid.

A precipitate is formed. Add potash, drop by drop, till the fluid is strongly alkaline.

> *The precipitate redissolves.* MORPHINE. Confirm by testing a portion of the original substance with iodic acid, and another portion with sulphuric acid &c.
>
> *The precipitate remains undissolved.* Presence of an alkaloid of Group II. or III. Pass on to 2.

2. Add to a second portion of the original solution 2 or 3 drops of dilute sulphuric acid, then a saturated solution of acid carbonate of sodium, until the acid reaction is just destroyed; well rub the inside of the vessel, and allow to stand for half an hour.

No precipitate is formed. Absence of narcotine and cinchonine. Pass on to 3.

A precipitate is formed. Narcotine, cinchonine, or perhaps quinine. To a portion of the original solution add ammonia in excess, then some ether, and shake.

> *The precipitate formed by the ammonia redissolves in the ether.* Narcotine or quinine. Test a fresh portion of the original solution with chlorine water and ammonia. If the solution turns green, QUININE is present; if yellowish-red, NARCOTINE is present. Confirm for the latter by testing with sulphuric acid mixed with nitric acid.
>
> *The precipitate formed by the ammonia does not redissolve in the ether.* CINCHONINE. Confirm by ferrocyanide of potassium.

3. Put a portion of the original substance, or of the residue remaining upon the evaporation of the solution, on a watch-glass, and treat with strong sulphuric acid. The solution obtained is

rose-colored, and becomes intensely red on addition of nitric acid. BRUCINE. Confirm with nitric acid and stannous chloride.

Yellow, and turns yellowish-red, blood-red, and finally crimson. VERATRINE.

Colorless, and remains so after long-standing. Add a fragment of chromate of potassium. A deep blue color indicates STRYCHNINE; no change indicates QUININE. Confirm the result by chlorine water and ammonia.

DETECTION OF THE NON-VOLATILE ALKALOIDS AND SALICINE WHEN PRESENT IN AN AQUEOUS SOLUTION TOGETHER.

1. To a portion of the solution add dilute potash, drop by drop, until the fluid acquires a scarcely perceptible alkaline reaction; stir, and allow to stand for some time. *If no precipitate is formed,* alkaloids are absent; pass on at once to 2. *If a precipitate is formed,* add some more potash, drop by drop, till the reaction is strongly alkaline. (If the precipitate is completely redissolved, morphine is the only alkaloid present; test the original solution with iodic acid, and with sulphuric acid, &c.; and then pass on to 2.) Filter.

The filtrate. Saturate with carbonic acid, add acid carbonate of sodium, boil nearly to dryness, and treat with water. If an insoluble residue remains, MORPHINE is indicated. Test with iodic acid and sulphuric acid, &c.

The precipitate. Wash with cold water, dissolve in a slight excess of dilute sulphuric acid, add acid carbonate of sodium till the acid reaction is neutralized, rub the sides of the vessel well, and allow to stand one hour. Filter.

The filtrate. Boil nearly to dryness, and treat with cold water. If an insoluble residue remains, wash it with water, dry on the water bath, and digest with absolute alcohol. Filter.

The filtrate. Evaporate to dryness and divide into three parts.

1. Test for BRUCINE with nitric acid and stannous chloride.
2. Test for VERATRINE with strong sulphuric acid.
3. Test for traces of quinine with chlorine water and ammonia. If brucine has been found, dissolve 3 in hydrochloric acid, add ammonia and

ether, allow to stand some time, evaporate the ethereal solution, and then examine the residue with chlorine water and ammonia.

The residue. Probably consists of STRYCHNINE. Test with sulphuric acid and chromate of potassium.

The precipitate. Wash with cold water, dissolve in a little hydrochloric acid, add excess of ammonia, then some ether, and shake. Separate the clear ethereal solution from the aqueous fluid and precipitate.

The ethereal solution. Evaporate, dissolve in a little hydrochloric acid, add water till the solution contains not more than ·5 per cent. of alkaloids, then acid carbonate of sodium to neutralization, and allow to stand some time. Filter.

The filtrate. Evaporate to dryness, and treat with water. If an insoluble residue remains, wash, dissolve in hydrochloric acid, and test for QUININE with chlorine water and ammonia.

The precipitate. Test with chlorine water and ammonia, and with sulphuric acid and nitric acid for NARCOTINE.

The aqueous fluid and precipitate. Collect the precipitate on a filter and examine it for CINCHONINE with ferrocyanide of potassium.

2. Mix a portion of the original solution with hydrochloric acid and boil for some time. A precipitate indicates SALICINE. Confirm by strong sulphuric acid.

REACTIONS OF ORGANIC ACIDS.

ORGANIC ACIDS, OF COMMON OCCURRENCE.

Acids which are precipitated by Nitrate of Silver in presence of Nitric Acid.

Hydrocyanic acid.
Hydroferrocyanic acid.
Hydroferricyanic acid.

Acids which are precipitated by Chloride of Calcium from Neutral Solutions.

Oxalic acid.
Tartaric acid.
Citric acid.

Volatile Acid.

Acetic acid.

Hydrocyanic Acid.

1. *Nitrate of silver* produces a white precipitate of cyanide of silver (AgCy), which is readily soluble in cyanide of potassium, rather difficultly soluble in ammonia, and insoluble in dilute nitric acid. The precipitate is decomposed by ignition, leaving silver and paracyanide of silver.

2. If *ferrous sulphate* and a little *ferric chloride* are added to a solution of a cyanide acidified with hydrochloric acid, no change takes place; but if potash is now added, a bluish-green precipitate forms, which consists of a mixture of Prussian blue [$Fe_4(Cy_6Fe)_3$] and ferroferric hydrate. Upon warming, and adding hydrochloric acid, the hydrate of iron dissolves, while the Prussian blue remains. In the presence of only traces of hydrocyanic acid the fluid simply appears green after

the addition of the hydrochloric acid, and it is only after long standing that a trifling blue precipitate separates.

3. If a liquid containing a little hydrocyanic acid or alkaline cyanide is mixed with enough yellow *sulphide of ammonium* to give it a yellow tint, then with a little ammonia, and the mixture is warmed in a porcelain dish until it has become colorless, and the excess of sulphide of ammonium is decomposed or volatilized, the fluid contains now sulphocyanide of ammonium. The reaction which takes place may be thus expressed—

$$(NH_4)_2S \,.\, S + HCy = NH_4CyS + NH_4HS.$$

On acidifying with hydrochloric acid (sulphuretted hydrogen should not be disengaged), and adding ferric chloride, the well-known blood-red color will be obtained. This reaction is very delicate. If an acetate is present the color is not obtained till more hydrochloric acid is added.

This test may be applied to insoluble cyanides as follows:—Fuse some hyposulphite of sodium in the loop of a platinum wire till the water of crystallization has escaped and the mass swells out, dip it in the substance, heat for a little time, removing it from the flame as soon as the sulphur begins to burn, and then dip the mass in a few drops of ferric chloride, mixed with a little hydrochloric acid. A permanent blood-red color will be produced. If the substance is heated too long the reaction fails, as the sulphocyanide of sodium formed is then destroyed again. This method is well adapted to distinguish chloride, bromide, or iodide of silver from cyanide of silver. (A. Fröhde.)

4. *Neither of the above methods will detect cyanogen in mercuric cyanide.* If the mercury is removed by sulphuretted hydrogen, hydrocyanic acid will be formed, and may be readily found. The solid salt may be heated in an ignition tube, when cyanogen will be evolved, and may be recognised by its odor and by its burning with a crimson flame.

Hydroferrocyanic Acid.

1. *Nitrate of silver* produces a white precipitate of ferrocyanide of silver (Ag_4Cy_6Fe), which is insoluble in nitric acid and ammonia, and dissolves in cyanide of potassium.

2. *Ferric chloride* produces a precipitate of Prussian blue [$Fe_4(Cy_6Fe)_3$].

3. *Sulphate of copper* produces a brownish-red precipitate of ferrocyanide of copper (Cu_2Cy_6Fe).

4. If an alkaline ferrocyanide is mixed with *hydrochloric acid,* and some *ether* is added, hydroferrocyanic acid separates where the two fluids meet in a crystalline form.

5. Insoluble ferrocyanides are decomposed by being boiled with potash ; ferrocyanide of potassium being formed, and the metallic oxide separated if it is insoluble in potash.

6. When ferrocyanides are heated with a mixture of three parts of strong sulphuric acid and one part of water till the free acid is driven off, they are decomposed, the cyanogen being evolved as hydrocyanic acid and the metals remaining as sulphates.

7. On projecting a ferrocyanide into fusing nitre, carbonic acid and nitrogen are evolved, while the metal is left behind in the form of oxide.

Hydroferricyanic Acid.

1. *Nitrate of silver* produces an orange colored precipitate of ferricyanide of silver (Ag_3Cy_6Fe) which is insoluble in nitric acid, but readily soluble in ammonia and cyanide of potassium.

2. *Ferric chloride* does not produce a blue precipitate.

3. *Ferrous sulphate* produces a blue precipitate of ferrous ferricyanide [$Fe_3(Cy_6Fe)_2$].

4. *Sulphate of copper* produces a yellowish green precipitate of ferricyanide of copper [$Cu_3(Cy_6Fe)_2$], which is insoluble in hydrochloric acid.

5. Insoluble ferricyanides are decomposed by being boiled with potash. The solution filtered from the metallic oxides contains only ferricyanide of potassium, or a mixture of ferrocyanide and ferricyanide of potassium.

6. By heating with a mixture of three parts of strong sulphuric acid and one part of water, the ferricyanides are decomposed like the ferrocyanides.

7. By fusing with nitre the ferricyanides are decomposed like the ferrocyanides.

Oxalic Acid.

1. *Chloride of calcium* produces a white pulverulent pre-

cipitate of oxalate of calcium (CaC_2O_4+aq. and occasionally CaC_2O_4+3aq.) which is as good as insoluble in water, almost insoluble in acetic acid and oxalic acid, readily soluble in hydrochloric acid and nitric acid. Ammonium salts do not in the least hinder the formation of the precipitate. Ammonia favors the precipitation of free oxalic acid. In very dilute solutions the precipitate takes some time to form.

2. *Lime water* produces the same precipitate.

3. *Sulphate of calcium* produces the same precipitate.

4. If a dry oxalate is heated with *strong sulphuric acid,* a mixture of carbonic acid and carbonic oxide escapes with effervescence. The carbonic oxide may be lighted as it escapes from the mouth of the test-tube. The sulphuric acid is not colored during this reaction.

5. If an oxalate is mixed with finely powdered binoxide of manganese (free from carbonic acid) and to the mixture a little water and strong sulphuric acid are added, an effervescence will occur due to the escape of carbonic acid, thus

$$K_2C_2O_4 + MnO_2 + 2H_2SO_4 = K_2SO_4 + MnSO_4 + 2H_2O + 2CO_2$$

Tartaric Acid.

1. *Chloride of calcium* added in excess to neutral tartrates throws down a precipitate of tartrate of calcium ($CaC_4H_4O_6$+4aq.). Ammoniacal salts retard the formation of this precipitate. Agitation of the fluid or friction on the side of the vessel promotes the separation. The precipitate is crystalline, or invariably becomes so after a short time. It dissolves in cold potash pretty free from carbonate to a clear fluid; but on boiling the solution, the tartrate of calcium separates in the form of a gelatinous precipitate, which redissolves on cooling.

2. *Lime-water* added in excess to neutral tartrates produces the same precipitate.

3. *Sulphate of calcium* added in excess produces a trifling precipitate in solutions of neutral tartrates of the alkali metals after the lapse of some time.

4. *Acetate of potassium and free acetic acid* produce a sparingly soluble precipitate of acid tartrate of potassium ($KHC_4H_4O_6$). The separation of the precipitate is greatly promoted by shaking or rubbing with a glass rod. The solu-

tion should be strong. The precipitate dissolves readily in mineral acids and alkalies; neither tartaric nor acetic acid increase its solubility in water.

5. *Acetate of lead* produces in solutions of tartaric acid and of neutral tartrates a precipitate of tartrate of lead ($PbC_4H_4O_6$), which is readily soluble in nitric acid and ammonia.

6. *Nitrate of silver* added to a solution of a neutral tartrate produces a white bulky precipitate of tartrate of silver ($Ag_2C_4H_4O_6$). If to the mixed precipitate and fluid a little ammonia is added to redissolve the precipitate nearly, but not completely, and heat is applied, a mirror of silver will be deposited on the test tube.

Citric Acid.

1. *Chloride of calcium* added in excess to neutral citrates produces a precipitate of citrate of calcium [$Ca_3(C_6H_5O_7)_2$], which is insoluble in potash, but readily soluble in chloride of ammonium. On boiling the solution of the precipitate in chloride of ammonium, the citrate of lime separates, but it is now insoluble in chloride of ammonium. If a solution of citric acid is mixed with excess of chloride of calcium, and then saturated with ammonia, a precipitate will not form in the cold till after long standing; but if the clear solution is boiled, citrate of calcium is immediately precipitated.

2. *Lime water* added in excess produces no precipitate in the cold. But if the solution is boiled for a long time with moderate excess of hot-prepared lime water, a white precipitate of citrate of calcium is produced, which disappears for the most part on cooling.

3. *Acetate of lead* added in excess to solution of citric acid produces a precipitate of citrate of lead [$Pb_3(C_6H_5O_7)_2$] which, after being washed, is readily soluble in ammonia.

4. *Nitrate of silver* added to neutral citrates of the alkali metals produces a precipitate of citrate of silver ($Ag_3C_6H_5O_7$). On treating this precipitate in the same manner as the tartrate no mirror is obtained.

Acetic Acid.

1. When acetates are distilled with *dilute sulphuric acid,* acetic acid passes over with the distillate.

2. When an acetate is heated with *strong sulphuric acid,* acetic acid escapes and may be recognised by its smell.

3. When an acetate is heated with a mixture of equal volumes of *strong sulphuric acid and pure spirit of wine,* acetic ether is evolved, which may be recognised by its very characteristic smell. The smell is most distinct upon shaking the mixture when it has cooled a little.

4. *Ferric chloride* added to an acetate produces a dark-red color from the formation of ferric acetate. If excess of acetate is present, and the fluid is boiled, it loses its color, the whole of the iron being precipitated as a basic acetate in the form of brownish-yellow flocks. Ammonia added to the red solution of ferric acetate, precipitates the whole of the iron as hydrate. Hydrochloric acid added to the red solution decolorizes it. (Here ferric acetate differs from ferric sulphocyanide.)

DETECTION OF POISONS IN BODIES, FOOD, &c.

Detection of Arsenic and other Metals.

PRELIMINARY SEARCH FOR UNDISSOLVED ARSENIOUS OXIDE.

Of all the combinations of arsenic this substance is most frequently used for criminal purposes. It dissolves in water very sparingly and also slowly, as moisture adheres to it with difficulty, hence when it has been swallowed the greater portion usually remains for some time in the undissolved state. This undissolved arsenious oxide is often readily separable by mechanical means, and when separated is most readily identified.

If the stomach or intestines have to be examined, empty the contents into a porcelain dish and turn the organs inside out. Search the inside coat for white, hard, sandy grains, which are often found firmly imbedded. Mix the contents in the dish as uniformly as possible, put aside one-third for use in case of accident and mix the rest in the dish with distilled water, by the aid of a glass rod ; let the mixture stand a little, then pour off the fluid, together with the lighter suspended particles, into another porcelain dish. Repeat this latter operation several times, if possible with the same fluid, pouring it from the second dish back into the first, &c. Finally, wash once more with pure water, preferably in a glass dish, remove the fluid as far as practicable, and try whether you can find in the dish white hard grains which feel gritty under the glass rod. If there are any such grains wash them, dry them on blotting paper, or in a watch-glass, weigh them, and test as directed p. 42, 3.

If you have to examine food, vomit, &c., treat it in the same manner as the contents of the stomach.

PRELIMINARY DIALYTIC EXAMINATION.

If you have failed in finding undissolved arsenious oxide, it may be worth while before proceeding to the regular method, to see if any poison can be dialysed out of the matter under examination.

For this purpose digest the two-thirds of the matter (with the addition, if occasion requires, of two-thirds of the organs cut small) for twenty-four hours at 32°, and then submit it to dialysis (see p. 21) also for twenty-four hours. Concentrate the dialysate by evaporation on the water bath, then acidify the greater portion with hydrochloric acid, treat with sulphuretted hydrogen, and proceed as directed p. 233, 2. If a soluble compound of arsenic or any other metal is present, the sulphide will be obtained nearly pure.

If arsenic is found here examine the rest of the concentrated dialysate to see whether it contains arsenious or arsenic acid.

THE REGULAR METHOD.*

The following method insures the detection of the minutest amount of arsenic, allows of its quantitative estimation, and permits at the same time the detection of all other metallic poisons.

1. *Decoloration and Solution.*

Evaporate the matter to a pasty consistence on the water bath,† add an amount of pure hydrochloric acid sp. gr. 1·12 about equal to or a little exceeding the weight of dry substances present, and sufficient water to give the mass the consistence of thin paste. (The quantity of hydrochloric acid should never exceed one-third of the entire liquid present.) Heat the dish on the water bath, adding every five minutes a small quantity (about 2 grm.) of chlorate of potassium with stirring, until the contents have a light-yellow color and are perfectly homogeneous and fluid; replace the evaporating water from time to time. Add a little more chlorate of potassium, and then allow to cool. When quite cold, transfer cautiously to a linen strainer or to a white filter, and allow the whole of the fluid to pass through. Heat the *filtrate* on the water bath with renewal of the evaporating water until the smell of chlorine has passed off. Wash the *residue* well with hot

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* This method is essentially the same as that published by the author and L. v. Babo in 1844, compare Annal. d. Chem. u. Pharm., 49, 308. The author has repeatedly employed it, and has always found it to work satisfactorily. Of course most scrupulous attention must be paid to the purity of the reagents.

† It may now weigh say 125—250 grm.

water, dry it, mark it I., and put it aside for subsequent examination. Evaporate the washings on a water bath to about 100 c.c. and add them, together with any precipitate that may have formed, to the principal filtrate.

2. *Precipitation with hydrosulphuric acid.*

Transfer the fluid obtained in 1, which amounts to three or four times the quantity of the hydrochloric acid used, to a flask, heat on a water bath to 70° and pass into it, for about twelve hours, a slow current of washed hydrosulphuric acid, then allow to cool, continuing the transmission of the gas all the while. Rinse the delivery tube with a little ammonia, acidify the rinsings and add them to the principal fluid, cover the flask lightly with paper and keep it in a moderately warm place (at about 30°) until the smell of hydrosulphuric acid has nearly passed off. Filter, and wash the precipitate until the washings are free from chlorine. Concentrate the filtrate and washings somewhat, transfer to a flask, add ammonia to alkaline reaction, then sulphide of ammonium, closely cork the flask, which should be nearly full, and reserve for subsequent examination according to 9 (p. 238).

3. *Purification of the precipitate produced by hydrosulphuric acid.*

The precipitate obtained in 2 contains the arsenic and other metals of Groups I. and II., as sulphides, and probably also organic matter and free sulphur. Thoroughly dry it with the filter in a small porcelain dish on a water bath, add pure fuming nitric acid (free from chlorine), drop by drop, till the mass is completely moistened, and then evaporate on the water bath to dryness. Moisten the residue uniformly all over with pure strong sulphuric acid, previously warmed, heat for two or three hours on the water bath, and finally in the air or paraffin bath at about 170°, until the charred mass is friable, and a small portion of it—to be returned afterwards to the mass—when mixed with water, and then allowed to subside, gives a colorless fluid. Should the fluid standing over the sediment show a brownish tint, or should the residue, instead of being friable, consist of a brown oily liquid, add to the mass some cuttings of Swedish filtering paper, and continue the application of heat.* Warm the residue on the water bath with a mixture of 1 part of hydrochloric acid and 8 parts of water, filter, wash the undissolved part thoroughly with hot water containing a little hydrochloric acid, and add the washings, after concentration if necessary, to the filtrate. Dry the washed carbonaceous residue, mark it II., and set it aside for subsequent examination.

4. *Second precipitation with hydrosulphuric acid.*

The clear and colorless, or at most somewhat yellowish, fluid obtained in 3 contains all the arsenic in the form of arsenious acid, and may contain also, mercury, copper, bismuth, cadmium, tin and antimony. Supersaturate a small portion cautiously with carbonate of ammonium, and observe whether a precipitate is produced, then acidify it with hydrochloric acid and return it to the rest. Transfer the fluid to a flask, and pass hydrosulphuric acid through it for some time. Rinse the delivery tube with a little ammonia if necessary, acidify the rinsings and add them to the principal fluid, cover the flask lightly with paper and keep it in a moderately warm place (at about 30°) until the smell of hydrosulphuric acid has nearly passed off.

> *If the precipitate is pure yellow.* Take a small portion of the fluid with the precipitate suspended in it, add some ammonia, and shake the mixture without warming. If the precipitate dissolves readily and (with the exception of a trace of sulphur) completely, and if carbonate of ammonium gave no precipitate before the passing of the hydrosulphuric acid, arsenic alone is present. If the precipitate does not dissolve, or if carbonate of ammonium has produced a precipitate, then there is reason to suspect the presence of another metal. In either case acidify the portion with hydrochloric acid and return it to the bulk of the fluid and precipitate.
>
> *If the precipitate is not pure yellow.* You have to assume the presence of metals other than arsenic.

Collect the precipitate on a filter and wash it thoroughly.

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* By attending to these directions you will always succeed in completely destroying the organic matter, without loss of any of the metals.

5. *Treatment of the pure hydrosulphuric acid precipitate when arsenic is the only metal present. Estimation of the weight of the arsenic.*

Treat the still moist precipitate on the filter with ammonia, and wash the filter thoroughly with diluted ammonia (nothing should remain undissolved, except some sulphur); evaporate the ammoniacal fluid in a small weighed porcelain dish on the water bath, dry the residue at 100° and weigh. If on subsequent reduction, it is found that this residue consists of pure arsenious sulphide, you can readily calculate it into arsenious oxide or into arsenic.

6. *Treatment of the pure hydrosulphuric acid precipitate when a metal other than arsenic is present. Separation of the arsenic and estimation of its weight.*

Perforate the point of the filter containing the still moist precipitate, wash the contents into a small flask, with the smallest possible quantity of water, add ammonia and sulphide of ammonium, and digest at a gentle heat. Filter if necessary, wash the residue, perforate the filter, rinse off the residue, mark it III., and reserve it for subsequent examination. Evaporate the filtrate and washings in a porcelain dish to dryness. Treat with pure fuming nitric acid (free from chlorine), nearly drive off the acid by evaporation, then add cautiously solution of carbonate of sodium in excess. Add now a mixture of 1 part of carbonate of sodium and 2 parts of nitrate of sodium, evaporate to dryness, and heat the residue very gradually to fusion, then allow to cool and extract with cold water. Filter if necessary, wash the residue with a mixture of equal parts of spirit and water, mark it IV., and reserve it for subsequent examination. Evaporate the washings to remove alcohol, and mix them with the solution. To the solution (which contains the arsenic as arseniate of sodium), add cautiously pure dilute sulphuric acid in excess, evaporate in a porcelain dish, and when strongly concentrated, add more sulphuric acid, to see whether the quantity first added was sufficient to expel all nitric and nitrous acids. Heat now cautiously till fumes of sulphuric acid begin to escape; allow to cool, add water, transfer to a small flask, heat to 70° and pass a slow current of washed hydrosulphuric acid for 6 hours.

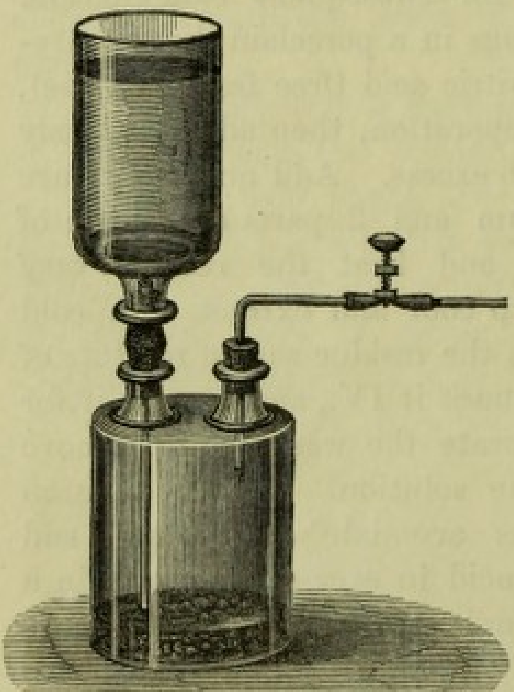
Allow to cool, continuing the transmission of the gas all the while. When the yellow precipitate has completely subsided, and the smell of the gas has nearly passed off, filter, wash the precipitate, dry it, extract the sulphur with bisulphide of carbon, dissolve in ammonia, and proceed with the solution according to 5, p. 235.

7. *Reduction of the sulphide of arsenic.*

The production of metallic arsenic from the sulphide, which may be regarded as the keystone of the whole process, demands the greatest care and attention. The method given p. 41, 2, which consists in fusing the sulphide mixed with cyanide of potassium and carbonate of sodium, in a slow current of carbonic acid, is the best and safest, as it is the most accurate, and as it obviates the possibility of any confusion between arsenic and antimony.

Do not use the whole of the sulphide at once, so that the process may be repeated several times if necessary. Should the quantity of sulphide be too minute to be divided, dissolve it in a few drops of ammonia, add a little carbonate of sodium, and evaporate with stirring on a water bath to dryness; you can then divide the residue into several portions. In such delicate experiments as the present the evolution flask figured p. 41 should be replaced by an apparatus which admits of the regulation of the current; such an apparatus is shown in the margin. Take care to drive all the air out of the reduction tube, and to give the current the proper degree of force before applying heat.

Fig. 6.



When the operation is finished, cut the reduction tube between the mirror and the mixture, and set aside the part containing the mirror. Put the other part into a test tube, add water, and let it stand some time; then filter. Add excess of hydrochloric acid to the *filtrate,* then hydrosulphuric acid, and observe whether a precipitate is formed. A trifling yellow precipitate will usually form if arsenic was present; if traces of antimony are present the precipitate will be orange-colored and insoluble in carbonate of ammonium. Examine the metallic *residue* which may be left for traces of tin and antimony, which might possibly be present. Should appreciable traces of either of these metals be found, proper allowance must be made for them in calculating the weight of the arsenic.

8. *Examination of the reserved residues for other metals of Groups I. and II.*

Residue I. This may contain chloride of silver, sulphate of lead, and possibly also stannic oxide. Incinerate it in a porcelain dish, burn the carbon with the aid of some nitrate of ammonium, extract the residue with water, dry the part left undissolved, and fuse it with cyanide of potassium in a porcelain crucible. When the fused mass is cold, extract it with water; warm the residue with nitric acid, and proceed as in the analysis of alloys.

Residue II. This may contain lead, mercury, tin, and possibly also bismuth and antimony. Heat for some time with aqua regia, filter, and wash the residue, at first with water containing a little hydrochloric acid. Treat the *filtrate* and washings with hydrosulphuric acid, and if a precipitate forms, examine it in the way in which a hydrosulphuric acid precipitate is usually examined. Incinerate the *residue,* fuse the ash with cyanide of potassium, allow to cool, extract with water, warm the residue with nitric acid, and proceed as in the analysis of alloys.

Residue III. Examine for metals of Group II., Division I., in the usual way.

Residue IV. This may contain tin, antimony and copper. Examine as usual.

9. *Examination of the reserved filtrate for metals of Group III., especially for zinc, chromium, and thallium.*

The fluid filtered from the hydrosulphuric acid precipitate in 2 and mixed with sulphide of ammonium should be filtered.

The precipitate. May contain ferrous sulphide, phosphate of calcium, and possibly also sulphide of zinc, sulphide of thallium, and hydrate of chromium. Wash it with water containing sulphide of ammonium, dissolve it by heating with hydrochloric acid and a little nitric acid, filter, and evaporate the filtrate with some sulphuric acid in a retort till the residue begins to be pasty. Test the distillate with iodide of potassium and in the spectroscope for thallium, a part of which may have escaped with the hydrochloric acid. Treat the residue in the retort with water, filter, add carbonate of sodium in excess, and then excess of cyanide of potassium (free from sulphide), heat for some time, and then filter.

- p_1. Reserve.
- f_1. Add sulphide of ammonium, and filter.
  - p_2. Test for thallium in the spectroscope.
  - f_2. Mix with p_1, evaporate with excess of sulphuric acid till the latter begins to escape, dilute, filter, precipitate with ammonia and sulphide of ammonium, and test the precipitate for zinc and chromium in the usual way.

The filtrate. May contain a portion, and indeed the whole of the chromium. Evaporate to dryness, incinerate, mix with 3 parts of chlorate of potassium and 1 part of carbonate of sodium, and project the mixture gradually into a red-hot crucible. If chromium is present the aqueous extract of the mass will be yellow.

Detection of Hydrocyanic Acid.

This method leaves the substance fit to be examined for metallic poisons and alkaloids.

In this case it is necessary to proceed with expedition, as hydrocyanic acid in contact with decaying animal or vegetable matter is liable to be soon decomposed.*

In the following process we separate the hydrocyanic acid by distilling the acidified matter. Now, if the matter contains the non-poisonous ferrocyanide or ferricyanide of potassium, they would be decomposed in this treatment, and hydrocyanic acid would be found in the distillate. Hence it is necessary first to ascertain whether either of these salts is present. To this end, stir a portion of the matter with water, filter, acidify with hydrochloric acid, and test one portion with ferric chloride, and another with ferrous sulphate. If no blue precipitate or color is produced in either case, you may safely proceed.

Test the reaction of the matter, if necessary, after stirring it with water. If it is not strongly acid, add solution of tartaric acid until it is. Then distil from a chloride of calcium bath, with the neck of the retort directed upwards and connected with a Liebig's condenser. Let the matter boil gently, and collect the distillate in a small weighed flask. When about 15 c.c. have passed over, replace the receiver by a somewhat larger flask, also weighed. Weigh the contents of the first receiver now, and proceed as follows :—

Test one quarter of the distillate according to p. 222,

Test another quarter of the distillate according to p. 223, 3. As the distillate may contain acetic acid, the precaution to be adopted in presence of acetates must not be forgotten.

If hydrocyanic acid has been found, and you wish to determine its quantity approximately, continue the distillation until the distillate is free from hydrocyanic acid. Add half of the contents of the second receiver to the remaining half of the contents of the first, and distil with some powdered borax,† leaving only a small residue in the retort. Add nitrate of silver to the distillate, and then nitric acid till strongly acid. Allow the precipitate to subside, collect it on a weighed filter, wash, dry at 100°, and weigh.

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* However, it is some time before the whole of the acid is lost. Thus, I succeeded in separating a notable quantity from the stomach of a man whose intestines were not handed to me till 36 hours after death. Again, a dog was poisoned with a small quantity of the acid, and the contents of the stomach, mixed with the blood, were left for 24 hours exposed to an intense summer heat, and then examined ; the acid was still detected.

† The object of the distillation with borax is to separate any hydrochloric acid that may be present.

By multiplying the quantity of cyanide of silver found by ·2017, you will find the corresponding amount of anhydrous hydrocyanic acid. And by multiplying this again by 2—as only half the distillate was used—you will find the total quantity of hydrocyanic acid in the matter examined.

Detection of Phosphorus.

PRELIMINARY EXAMINATION.

1. Notice whether the matter smells of phosphorus, or is luminous in the dark, taking care at the same time to bring it thoroughly into contact with the air by stirring or shaking.

2. If phosphorus has not been indicated in 1, put a little of the matter into a flask, suspend in the latter, by the aid of a loose cork, a slip of filtering paper moistened with nitrate of silver, and heat to 30° or 40°. If the paper does not turn black, even after some time, no unoxidized phosphorus is present, and it will be useless to proceed to the regular examination. If, on the other hand, the paper turns black, this is no positive proof of the presence of unoxidized phosphorus, since hydrosulphuric acid,* formic acid, putrifying substances, &c., will produce this effect.

THE REGULAR EXAMINATION.

For this, one of the two following methods may be used.

Method I.†

Mix the matter with water and some sulphuric acid, and distil slowly in a flask with a Liebig's condenser. If the matter contains phosphorus, a strong luminosity, usually a luminous ring, will appear in the dark in that part of the inner tube of the condenser which is first cooled. If you take for distillation 150 c.c. of a mixture containing only 1·5 mgrm. of phosphorus, and accordingly only 1 part of phosphorus in 100,000 parts, you may distil over 90 c.c.—which will take at least half an hour—without the luminosity ceasing. Mitscherlich, in one of his experiments, stopped the distillation

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* To ascertain whether the blackening is caused by hydrosulphuric acid, you may test with a strip of paper moistened with acetate of lead.

† E. Mitscherlich, Journ. f. prakt. Chem., 66. 238.

after half an hour, allowed the flask to stand uncorked for a fortnight, and then recommenced the distillation; the luminosity was as strong as at first.

In the presence of substances which prevent the luminosity of phosphorus, such as alcohol, ether, or oil of turpentine, no luminosity is observed so long as these substances continue to distil over. In the case of ether and alcohol, however, this is soon effected, and the luminosity accordingly very speedily makes its appearance; but it is otherwise with oil of turpentine, which completely prevents the manifestation of this reaction.

After the termination of the process, globules of phosphorus are found at the bottom of the receiver. Mitscherlich obtained from 150 c.c. of a mixture containing 20 mgrm. of phosphorus so many globules of that body, that one-tenth of them would have been amply sufficient to demonstrate its presence.

Method II.

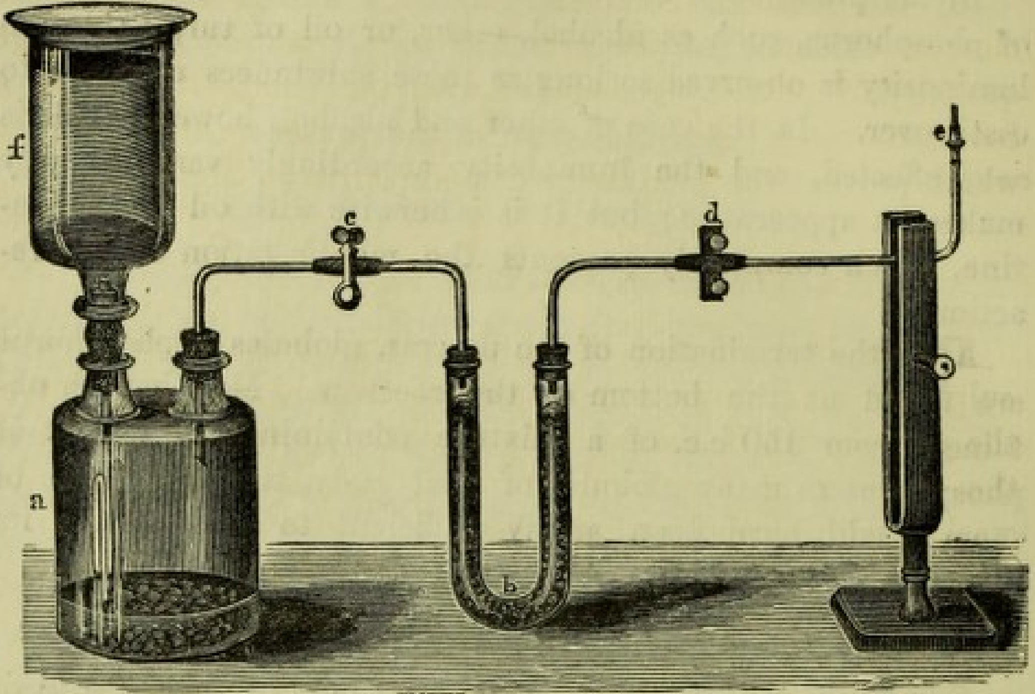
Put the matter in a flask, add water if necessary, and then some dilute sulphuric acid to acid reaction. Connect the flask on one side with a carbonic acid apparatus by means of a tube leading to the bottom of the flask, and on the other side with a U tube containing solution of nitrate of silver. Pass a slow stream of washed carbonic acid. When the air is driven out, heat the flask on a water-bath gently, and continue the passing of the gas and the warming of the flask for several hours. If free phosphorus is present, it will volatilize unoxidized in the current of carbonic acid, and will be converted in the U tube into black insoluble phosphide of silver, and into phosphoric acid. If no precipitate forms you may safely conclude that no unoxidized phosphorus is present. If a precipitate does form, it must not be taken as an indication of phosphorus, but both it and the fluid in which it is suspended must be examined as follows :—

Filter through a filter washed with nitric acid and water, and wash the precipitate with dilute nitric acid and water.

The precipitate. For the examination of this we require the following apparatus.

a is for evolving hydrogen, *b* contains pumice moistened with strong potash, *c* is a common clip, *d* a screw-clip, and *e* a platinum jet,* which is kept cool by means of moistened wool or cotton.

FIG. 7.



To ascertain whether the hydrogen is free from phosphuretted hydrogen, after the air has been driven out, close *c* until the fluid in *a* has ascended to *f*. Close *d*, open *c*, and regulate *d* so as to obtain a flame of suitable size. If the flame, viewed in a dark place, is colorless, showing no trace of a green cone in the centre, and no emerald coloration if directed upon a piece of porcelain (as in Marsh's test), then the hydrogen may be considered pure.

Rinse the precipitate with water into *f*, take care that every particle of it reaches *a*, allow *a* to fill with gas, and then light it again and observe the flame. If the precipitate contains even a trace of phosphide of silver, the green cone in the centre of the flame, and the emerald coloration when the flame impinges on porcelain, will be visible.

The filtrate. Precipitate the silver by hydrochloric acid, filter through a paper well washed with acid and water, evaporate off the hydrochloric acid, take up with nitric

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* The platinum jet is indispensable for the production of a colorless hydrogen flame.

acid, and test for phosphoric acid with molybdate of ammonium.

By this method, Neubauer and the author obtained the clearest evidence of the presence of phosphorus in a large quantity of putrid blood mixed with the composition from a common match; and this even in the presence of substances which prevent the luminosity of phosphorus in Method I.

ESTIMATION OF THE PHOSPHORUS.

This is best effected by distilling the mass, acidified with sulphuric acid, in an atmosphere of carbonic acid. Most of the phosphorus, if there is any quantity, will be obtained in the receiver in the form of small globules, which may be washed with alcohol and weighed.

Detection of Alkaloids.

Some alkaloids may be more or less liable to decomposition in the organism. There seems to be no doubt for instance that morphine is thus decomposed, as the following experiment shows. A rabbit had ·1 grm. of hydrochlorate of morphine given to it, and was killed 3½ hours afterwards; no morphine was found in the urine, brain, and spinal marrow, only very little in the blood, a little in the stomach and small intestines, more in the other intestines.

* STAS'S METHOD.

Stas enumerates the following alkaloids as discoverable by this method—conine, nicotine, aniline, picoline, petinine, morphine, codeine, brucine, strychnine, veratrine, colchicine, delphine, emetine, solanine, aconitine, atropine, hyoscyamine.

If you have to look for alkaloids in the contents of the stomach or intestines or in articles of food, heat the matter with twice its weight of strong alcohol acidified with 1 or 2 grm. of tartaric or oxalic acid at a temperature of 70° or 75°. When quite cold, filter, and wash the undissolved part with strong alcohol, adding the washings to the filtrate.

If you have to deal with the heart, liver, lungs or similar organs, cut them small, moisten with acidified alcohol, press, and repeat the operation, until the soluble parts are completely extracted; collect the fluids obtained and filter.

Concentrate the alcoholic fluid at a temperature not exceeding 35°, and if no insoluble matter separates, continue to evaporate nearly to dryness. Conduct this process either under a bell-glass over sulphuric acid, or in a tubulated retort, through which a current of air is passed. If fatty or other insoluble matters separate in the process of concentration, pass the concentrated fluid through a moistened filter, and evaporate the filtrate nearly to dryness, conducting the process as above either under a bell-glass or in a retort.

Digest the residue with cold absolute alcohol, filter, wash the insoluble part with alcohol, and let the alcoholic solution evaporate in air or in vacuo; dissolve the acid residue in a little water and add acid carbonate of sodium as long as effervescence ensues.

Add to the mixture four or five times its volume of pure ether, free from oil of wine, and shake; then allow to stand at rest.

Transfer a small quantity of the supernatant ether to a watch-glass and allow it to evaporate spontaneously. If oily streaks are left, which gradually collect into a drop, and emit, upon being gently heated, a disagreeable, pungent and stifling odor, there is reason to infer the presence of a volatile base. If a solid residue or a turbid fluid with solid particles suspended in it is left, a non-volatile base is probably present. In the latter case the base may emit a disagreeable animal smell, but not a pungent odor, as is the case with volatile bases. If no residue is left, to the rest of the fluid add some potash, and shake with repeatedly renewed ether which will now dissolve the base.

Now proceed as follows according to the results of this preliminary examination.

If there is reason to infer the presence of a volatile base.

To the mixture of aqueous solution and ether add 1 or 2 c.c. of strong potash, shake, allow to stand at rest, pour off the supernatant ether, and treat the watery fluid with fresh ether three or four times, till the last portion poured off leaves no residue on evaporation. Mix the ethereal solution with dilute sulphuric acid until the fluid after being well shaken manifests strongly acid reaction; allow the mixture to stand at rest, decant the supernatant ether, and treat the aqueous fluid once more in the same way.

> *The aqueous solution* contains sulphates of ammonia, nicotine, aniline, picoline, and petinine if they are present, and the greater part of the conine. Mix it with strong potash in excess, and treat with ether which will redissolve the liberated bases. Decant the ether, and leave it to spontaneous evaporation at the lowest possible temperature; then place the dish in vacuo over sulphuric acid. In this process the ether and ammonia escape, leaving the volatile alkaloid in the pure state.
>
> *The ethereal solution* contains the animal matters which it has removed from the alkaline fluid. It leaves therefore upon spontaneous evaporation, a faint yellow residue of nauseous odor, which contains also some sulphate of conine if that base was present.

If there is reason to infer the presence of a non-volatile base.

Pour off the ether from the mixture of aqueous solution and ether, add a few drops of alcohol to it, and leave it to spontaneous evaporation. If this fails to give the base in a distinctly crystalline form and sufficiently pure, add a few drops of water slightly acidified with sulphuric acid, which will usually serve to separate the residue into a fatty portion adhering to the dish, and an acid aqueous solution, which contains the base as an acid sulphate. Decant or filter, wash with a little slightly acidified water, and evaporate the solution to a considerable extent, under a bell-glass over sulphuric acid. Mix the residue with a very strong solution of pure carbonate of potassium, treat the mixture with absolute alcohol, decant, and let the alcoholic fluid evaporate, which will generally leave the base in a state of almost perfect purity.

OTTO'S MODIFICATIONS OF STAS'S METHOD.

1. In the method just described, the morphine which may be present will only pass into the ethereal solution if, after the addition of the acid carbonate of sodium, the solution is immediately shaken with ether, and the ether is then quickly

decanted. But if the operation of shaking with ether is delayed, so as to give the morphine time to crystallize, the crystals will deposit, being almost absolutely insoluble in ether. Again, if the morphine has dissolved in the ether, and the solution is allowed to stand some time, the morphine will separate in small crystals on the side of the vessel.

Hence it is always probable that the morphine will remain wholly or in part undissolved by the ether, and the alkaline fluid, after being extracted with ether, should be mixed with potash to dissolve separated morphine, evaporated to remove ether still present, mixed with chloride of ammonium, and allowed to stand exposed to the air to allow the morphine to crystallize.

2. For the process given for the detection of non-volatile alkaloids, Otto substitutes the following:—

Let the ethereal solution evaporate, dissolve the residuary impure alkaloid in a little water mixed with sulphuric acid, and shake the solution repeatedly with ether, which will remove the foreign organic substances present, and leave the acid sulphate of the alkaloid unaffected. Mix now the aqueous solution with carbonate of sodium in excess, shake repeatedly with ether (to dissolve the liberated alkaloids), and let the ethereal solution evaporate, when the alkaloids held in solution by the ether will be left in a very pure state, and to a great extent in the crystalline form.

3. But what Otto recommends most is the treatment of the alkaloid in the form of salt, with ether, before the addition of the carbonate of sodium.

Shake the aqueous solution, which contains the tartrate or oxalate of the alkaloid, with ether repeatedly, so long as the ether becomes colored and yields a residue on evaporation. Then add the acid carbonate of sodium, shake with ether, and proceed as directed. Upon evaporating the ether, the alkaloid is now left in a very pure state.

JANSSENS'S MODIFICATION OF STAS'S METHOD.

Janssens says that Stas's process, even with Otto's modification, does not give the strychnine sufficiently pure, especially when substances are present which are soluble in alcohol, water, ether, and dilute sulphuric acid. He recommends the following modification :—

Having exhausted the matter with acidified alcohol, evaporated, taken up with alcohol, and evaporated to dryness, dissolve in 25—50 c.c. of water, and then add finely powdered acid carbonate of sodium till the fluid is only acid through carbonic acid. If any precipitate forms, filter it off immediately. The strychnine remains dissolved in the carbonic acid, and does not precipitate till the filtrate is boiled and partially evaporated. When it has been collected on a filter and washed, dissolve it in a small quantity of very dilute sulphuric acid (1 of strong acid, 200 of water), add carbonate of potassium in excess, shake repeatedly with six times the volume of ether, and allow the latter to evaporate.

METHOD OF L. v. USLAR AND J. ERDMANN.

This may be considered to be an improvement upon Stas's method as regards non-volatile alkaloids, especially morphine, while Stas's method deserves the preference for volatile alkaloids. This method is the same in principle as Stas's with the exception that amyl alcohol is substituted for ether.

Mix the matter with water, if necessary, to the consistence of a thin paste, acidify slightly with hydrochloric acid, digest for one or two hours at a temperature between 60° and 80°, and strain through linen moistened with water. Extract the residue with hot water acidified with hydrochloric acid, mix the two solutions, supersaturate with ammonia, and evaporate to dryness with addition of pure sand, which will enable you to reduce the residue to powder. Boil the powder repeatedly with amyl alcohol, to extract the whole of the alkaloid from it, filtering the extracts hot.

The filtrate, which is mostly colored yellow, holds, besides the alkaloid, fatty and coloring matters in solution. To remove these latter, transfer to a cylinder, add ten volumes of almost boiling water, acidified with hydrochloric acid, and vigorously shake the mixture for some time. The hydrochlorate of the alkaloid passes into the water, while the fatty and coloring matters remain dissolved in the amyl alcohol. Remove the latter with a pipette, shake the acid solution repeatedly with fresh quantities of amyl alcohol, until the fatty and coloring matters are completely removed. Now concentrate by evaporation, add ammonia in slight excess, then some hot amyl alcohol, and shake vigorously. Allow to stand, draw off the upper layer (the solution of the alkaloid in amyl alcohol), and treat the residual fluid once more with hot amyl alcohol. Finally evaporate off the amyl alcohol on a water bath, when the alkaloid will generally be left sufficiently pure.

Should the residue, however, not be perfectly white, redissolve it in hydrochloric acid, shake the solution with amyl alcohol, draw off the latter, supersaturate with ammonia, shake again with amyl alcohol, then draw it off and evaporate on a water bath. Before proceeding to the decisive reactions, treat a portion of the alkaloid with a few drops of strong sulphuric acid; if a brown color is produced the purification must be repeated.

Uslar and Erdmann have isolated and detected by this method very minute quantities of alkaloids, *e.g.*, 5 mgrm. of hydrochlorate of morphine, 1 drop of nicotine, 9 mgrm. of strychnine, mixed with 2 or 3 pounds of contents of the stomach. Erdmann calls particular attention to the fact that he succeeded in separating strychnine and morphine from quite putrid intestines of poisoned animals twenty or thirty days after death.

METHODS FOR DETECTING STRYCHNINE, DEPENDING ON THE USE OF CHLOROFORM.

1. *After Rodgers and Girdwood.*

Digest with dilute hydrochloric acid (1 of strong acid to 10 of water), and filter. Evaporate the filtrate on the water-bath to dryness, extract the residue with spirit of wine, evaporate the solution, treat the residue with water, and filter. Supersaturate the filtrate with ammonia; add 10 c.c. of chloroform, and shake. Transfer the chloroform to a dish with a pipette, evaporate on the water-bath, moisten the residue with strong sulphuric acid to carbonize the foreign organic substances, treat with water after the lapse of several hours, and filter. Supersaturate the filtrate again with ammonia, and shake it with about 3 c.c. of chloroform. Repeat the same operation until the residue left on evaporation of the chloroform is no longer blackened by sulphuric acid. Finally transfer the pure chloroform solution, drop by drop by means of a capillary tube to the same spot on a heated porcelain dish, letting it evaporate, then test the residue with sulphuric acid and chromate of potassium. Rodgers and Girdwood succeeded in detecting by this method ·03 mgrm. of strychnine.

2. *After Prollius.*

Boil twice with spirit of wine, mixed with some tartaric acid, evaporate at a gentle heat, filter the watery solution remaining through a moistened filter, add ammonia in slight excess, then 1 c.c. of chloroform, shake, wash the chloroform thoroughly by decanting and shaking with water, then mix it with 3 parts of spirit of wine, and allow to evaporate. If there is any notable quantity of strychnine present, it will be obtained in crystals.

3. *After R. P. Thomas.*

This method includes the detection of morphine.

Acidify with pure acetic acid* slightly, and digest for several hours at a gentle heat, then strain, press, filter, add potash in good excess, and shake with chloroform. Separate the chloroform, wash it from potash, and evaporate; the strychnine will be found in the residue. The morphine remains in the potash, and may be precipitated gradually by chloride of ammonium.

METHOD OF DETECTING STRYCHNINE BY MEANS OF ANIMAL CHARCOAL.

Graham and Hofmann employed the following method for detecting strychnine in beer:—

Shake 2 oz. of animal charcoal in half a gallon of the beer; let the mixture stand for 12 or 24 hours, with occasional shaking, filter, wash the charcoal twice with water, then boil for half an hour with 8 oz. of spirit of wine of 80—90 per

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* Acetic acid is recommended because the tannates of strychnine and morphine are soluble in it.

cent., avoiding loss of alcohol by evaporation. Filter hot and distil the filtrate; add a few drops of potash to the residual watery fluid, shake with ether, and when the latter has separated, decant it. Upon the spontaneous evaporation of the ether the alkaloid will be left sufficiently pure.

Macadam employed the same method in his numerous experiments to detect strychnine in the bodies of dead animals. He treated the finely-divided matter with a dilute solution of oxalic acid in the cold, filtered through muslin, washed with water, heated to boiling, filtered still warm from the coagulated albuminous substances, shook with charcoal, and proceeded in the manner first described. The residue left by the evaporation of the alcoholic solution was generally at once fit to be tested for strychnine; where it was not, the process of treating with oxalic acid, shaking with charcoal, &c., was repeated.

SEPARATION BY DIALYSIS.

This process may be advantageously employed for the separation of crystallizable alkaloids from the contents of the stomach, &c. The matter should be acidified with hydrochloric acid before being dialysed. The dialysate may be concentrated by evaporation, and the alkaloid precipitated, and, if necessary, purified as before described.

TABLES.

LIST OF TABLES.

ANALYTICAL TABLES.

TABLE OF SOLUBILITY.

ANALYTICAL TABLES.

PRELIMINARY EXAMINATION.

Experiments.	Results.	Inferences.
Heat substance in ignition-tube	A sublimate	$S.NH_4.Hg.As.$
If a sublimate other than S— Heat substance with KHO in test tube	NH_3	NH_4
Also heat substance with dry Na_2CO_3 in ignition-tube	Globules of Hg	Hg
	As mirror	As
Heat substance with Na_2CO_3 on charcoal before the blow-pipe	Metallic globules Mal.	Pb.Ag.
	Metallic globules Brit.	Sb.Bi.
	Garlic odor	As
If the substance is colored, heat in the borax bead	The cold bead is—	
	blue,	Co.Cu.
	green,	Cr
	dark-yellow or bottle-green,	Fe
	reddish-brown,	Ni
	amethyst-red,	Mn
Heat substance on platinum wire in Bunsen flame.	The flame is colored—	
	azure,	Cu
	green,	$Cu . B_2O_3$
	yellowish-green,	Ba
	crimson,	Sr
	red,	Ca
	yellow,	Na
	violet,	K

TABLES FOR THE DETECTION OF METALS IN SOLUTIONS OF MIXED SUBSTANCES.

General Table.

Add HCl and Filter				
Precipitate.	*Filtrate.* Pass H_2S and filter.			
	Precipitate.	*Filtrate.* Add NH_4Cl, NH_4HO and $(NH_4)_2S$ and filter.		
		Precipitate.	*Filtrate.* Add $(NH_4)_2CO_3$ and filter.	
			Precipitate.	*Filtrate.*
AgCl HgCl $PbCl_2$ Wash and examine according to Table I.	PbS HgS Bi_2S_3 CuS CdS SnS SnS_2 Sb_2S_3 As_2S_3 Wash and examine according to Table II.	NiS CoS FeS MnS ZnS CrH_3O_3 AlH_3O_3 Certain salts of Ba Sr Ca Mg and SiO_2 Wash and examine according to Table III.	$BaCO_3$ $SrCO_3$ $CaCO_3$ Wash and examine according to Table IV.	Mg K Na NH_4 Examine according to Table V.

TABLE I.

<table>
<tr><td colspan="3">Treat on the filter with hot water.</td></tr>
<tr><td rowspan="2"><i>Filtrate.</i>
Add H_2SO_4.
White p. = Pb.</td><td colspan="2"><i>Residue.</i> Treat on the filter with NH_4HO.</td></tr>
<tr><td><i>Filtrate.</i>
Add excess of HNO_3.
White p. = Ag.</td><td><i>Residue</i>
is black = Hg′</td></tr>
</table>

TABLE II.

<table>
<tr><td colspan="6">Warm with $(NH_4)_2S$ and filter.</td></tr>
<tr><td colspan="5"><i>Residue.</i> Wash, boil with strong HNO_3, dilute and filter.</td><td rowspan="4"><i>Filtrate.</i> Add HCl in excess, filter, wash the p. and dry it. Divide in 2 parts.
1. Boil with strong HCl, dilute, filter into platinum crucible lid, add Zn. (black stain = Sb). Remove Zn, boil p. with strong HCl, filter, and to f. add $HgCl_2$. p. = Sn.
2. Fuse with KCy + Na_2CO_3 in ignition tube. Mirror = As.</td></tr>
<tr><td colspan="2" rowspan="2"><i>Residue.</i>
Divide in 2 parts.
1. Dissolve in HCl + $KClO_3$, and add $SnCl_2$. A grey or white p. = Hg″.
2. Fuse with KCy + Na_2CO_3. If metallic globules, wash them, and treat with HNO_3.</td><td colspan="3"><i>Filtrate.</i> Add H_2SO_4 dil., evaporate on water-bath till HNO_3 expelled, take up with H_2O and filter.</td></tr>
<tr><td rowspan="2"><i>Residue.</i>
= Pb.</td><td colspan="2"><i>Filtrate.</i> Add excess NH_4HO, warm and filter.</td></tr>
<tr><td><i>p.</i>
Wash, boil with strong HCl, pour off acid, add H_2O and then H_2S. A yellow p. = Sn.</td><td><i>f.</i>
Add H_2SO_4.
p. = Pb.</td><td><i>Precipitate.</i>
Wash, dissolve in watch-glass in least quantity of HCl, and add H_2O. Milkiness = Bi.</td><td><i>Filtrate.</i> (If blue, Cu is present). Add H_2S, wash p., boil it with H_2SO_4 dil., filter off CuS, and add H_2S. A yellow p. = Cd.</td></tr>
</table>

TABLE III.

When Salts and Silica are absent.

<table>
<tr><td colspan="4">Treat with cold dilute HCl, and filter.</td></tr>
<tr><td rowspan="3"><i>Residue.</i> Wash, test a portion in borax bead.
A blue bead = Co
A red bead = Ni
If a blue bead, dry the filter, incinerate, dissolve ash in HCl + HNO_3, nearly neutralize with KHO, add KNO_2 and $H\bar{A}$ till acid, allow to stand, filter off yellow p. To f. add KHO, filter and test p. in borax bead for Ni.</td><td colspan="3"><i>Filtrate.</i> Add strong HNO_3, boil, nearly neutralize with KHO. When cold add excess $BaCO_3$, and filter.</td></tr>
<tr><td rowspan="2"><i>Precipitate.</i> Wash and divide into 3 parts.
1. Dissolve in HCl, and add KCNS. A red color = Fe.
2. Boil with Na_2OCl_2, and filter. A yellow f. = Cr.
3. Boil with KHO, filter, to f. add NH_4Cl, and warm. p. = Al.</td><td colspan="2"><i>Filtrate.</i> Add H_2SO_4, filter off $BaSO_4$, evaporate to small bulk, add excess KHO, and filter.</td></tr>
<tr><td><i>Precipitate.</i>
Test in Na_2CO_3 bead for Mn.</td><td><i>Filtrate.</i>
Add H_2S. A white p. = Zn.</td></tr>
</table>

TABLE III.

When Salts and Silica are present.

Treat with cold dilute HCl and filter.

Residue. Wash, test a portion in microcosmic bead.

A skeleton = **SiO_2**.

A blue bead = **Co.**

A yellow bead = **Ni.**

If a blue bead, dry, filter, incinerate, dissolve ash in HCl + HNO_3, nearly neutralize with KHO, add KNO_2, and $H\bar{A}$ till acid, allow to stand, filter off yellow p. To f. add KHO, filter and test p. in borax bead for **Ni.**

Filtrate. Boil to expel H_2S, filter, if necessary, and divide in 2 parts.

1. Add dilute H_2SO_4 and filter. Wash and examine the *precipitate* for **Ba** and **Sr.** Mix the *filtrate* with three volumes of alcohol, collect the p., dissolve in H_2O, and test for **Ca** with $(NH_4)_2\bar{O}$.

2. Add strong HNO_3 and boil. Test a small portion for **Fe** with KCNS. To the rest add $FeCl_3$ till a drop gives a yellow p. with NH_4HO, evaporate to small bulk, add H_2O, nearly neutralize with KHO, add excess $BaCO_3$, allow to stand, filter.

Precipitate. Wash and divide in two parts.

1. Boil with Na_2OCl_2 and filter. A yellow f. = **Cr.**

2. Boil with KHO and filter. To f. add NH_4Cl, and boil; p. may be Al_2O_3 and SiO_2. Test it for **SiO_2** in microcosmic bead. If SiO_2 present, ignite rest of p., fuse with $KHSO_4$, treat with HCl, and filter; to f. add NH_4HO. A p. = **Al.**

Filtrate. Add HCl, boil to expel CO_2, add NH_4HO and $(NH_4)_2S$. Filter.

Precipitate. Wash with H_2O and a little $(NH_4)_2S$, treat with $H\bar{A}$ and filter.

Residue. Wash, treat with dilute HCl, and filter.* Treat with HNO_3, evaporate, add KHO, boil, and filter.* To f. add $(NH_4)_2S$. A white p. = **Zn.**

Filtrate. Add KHO, boil, filter, and test p. for **Mn.**

Filtrate. Add H_2SO_4, boil, filter off $BaSO_4$, add excess NH_4HO, then $(NH_4)_2\bar{O}$, filter off $Ca\bar{O}$, and test for **Mg.**

---

* If a residue test for Ni and Co.

TABLE IV.

Dissolve in HCl, evaporate to dryness on the water bath. Dissolve a portion of the residue in a little water, to the solution add $CaSO_4$, and allow to stand.

No precipitate is formed. Dissolve rest of residue in water, and add $(NH_4)_2\bar{O}$. A p.=**Ca.**

A precipitate is formed after some time.=**Sr.** Dissolve rest of residue in water, boil with $(NH_4)_2SO_4$ and NH_4HO for some time, filter off $SrSO_4$, and to f. add $(NH_4)_2\bar{O}$. A p.=**Ca.**

A precipitate is formed immediately.=**Ba.** Digest rest of residue with alcohol, powdering it in the dish with a pestle, filter off the $BaCl_2$, to f. add H_2SO_4, and filter. Boil p. with $(NH_4)_2SO_4$ and NH_4HO for some time and filter.

Residue. Test for **Sr** on platinum wire in Bunsen flame.	*Filtrate.* Dilute and add $(NH_4)_2\bar{O}$. A p.=**Ca.**

TABLE V.

To a portion of the solution add Na_2HPO_4, stir well, and allow to stand. A crystalline p.=**Mg.**

If Mg is absent. Evaporate rest of solution to dryness, ignite on piece of porcelain till white fumes cease, dissolve the residue in the least quantity of water, filter if necessary into a watch-glass, and test for K and Na as below.

If Mg is present. Evaporate rest of solution to dryness, ignite till white fumes cease, warm residue with a little water, add milk of lime till alkaline, boil, filter, to f. add $(NH_4)_2CO_3$, warm gently, filter, evaporate f., ignite, dissolve residue in least quantity of water, add a drop of HCl, pour solution into watch-glass, and test for K and Na as below.

Dip a clean platinum wire into the solution, and hold it in Bunsen flame, a yellow color=**Na.** Then add $PtCl_4$ to the solution and stir; a yellow p.=**K.** If no p., evaporate to dryness on water bath, add a drop or two of water, and observe whether yellow powder remains undissolved.

Warm the original substance with KHO in a test tube. A smell of NH_3=**NH_4**.

TABLE OF SOLUBILITY.

W— Soluble in water. *A*— Insoluble in water, soluble in acids. *I*— Insoluble in water and acids.

Capitals indicate substances of comparatively common occurrence.

	Aluminium.	Ammonium.	Antimony.	Barium.	Bismuth.	Cadmium.	Calcium.	Chromium.	Cobalt.	Copper.	Dyad Iron.	Triad Iron.	Lead.	Magnesium.	Manganese.	Monad Mercury.	Dyad Mercury.	Nickel.	Potassium.	Silver.	Sodium.	Strontium.	Dyad Tin.	Tetrad Tin.	Zinc.
Arseniate .	*a*	*w*	*a*	*a*	*a*		*a*	*a*	*a*	*a*	*a*	*a*	*a*	*a*	*a*	*a*	*a*	*a*	*W*	*a*	*W*	*a*		*a*	*a*
Arsenite .		*w*	*a*	*a*			*a*		*a*	*A*	*a*	*a*	*a*	*a*	*a*	*a*	*a*	*a*	*W*	*a*	*w*	*a*			
Borate . .	*a*	*w*		*a*	*a*	*w—a*	*a*	*a*	*a*	*a*	*a*	*a*	*a*	*w—a*	*a*			*a*	*w*	*a*	*w*	*a*	*a*		*a*
Bromide .	*w*	*W*	*w—a*	*w*	*w—a*	*w*	*w*	*w & i*	*w*	*w*	*w*	*w*	*w—i*	*w*	*w*	*a—i*	*w*	*w*	*W*	*i*	*W*	*w*			*w*
Carbonate .		*W*		*A*	*A*	*a*	*A*		*A*	*A*	*A*		*A*	*A*	*A*	*a*	*a*	*A*	*W*	*a*	*W*	*A*			*A*
Chlorate .	*w*	*w*		*W*	*w*	*w*	*w*	*w*	*w*	*w*	*w*	*w*	*w*	*w*	*w*	*w*	*w*	*w*	*W*	*w*	*w*	*w*	*w*		*w*
Chloride .	*w*	*W*	*W—A*	*W*	*W—A*	*W*	*W*	*W & I*	*W*	*W*	*W*	*W*	*W—I*	*W*	*W*	*A—I*	*W*	*W*	*W*	*I*	*W*	*W*	*W*	*W*	*W*
Chromate .		*w*	*a*	*a*	*a*	*a*	*w—a*	*a*	*a*	*w*		*w*	*A—I*	*w*	*w*	*a*	*w—a*	*a*	*W*	*a*	*w*	*w—a*	*a*		*w*
Fluoride .	*w*	*W*	*w*	*a—i*	*w*	*w—a*	*A—I*	*w*	*w—a*	*a*	*w—a*	*w*	*a*	*a—i*	*a*		*w—a*	*w—a*	*w*	*w*	*w*	*a—i*	*w*	*w*	*w—a*
Iodide . .	*w*	*W*	*w—a*	*w*	*a*	*W*	*w*	*w*	*w*	*w*	*W*	*w*	*W—A*	*w*	*w*	*A*	*A*	*w*	*W*	*i*	*w*	*w*	*w*	*w*	*w*
Nitrate . .	*w*	*W*		*W*	*W*	*w*	*w*	*W*	*W*	*W*	*w*	*w*	*W*	*w*	*w*	*W*	*W*	*w*	*W*	*W*	*W*	*W*			*w*

TABLE OF SOLUBILITY—*continued.*

	Aluminium.	Ammonium.	Antimony.	Barium.	Bismuth.	Cadmium.	Calcium.	Chromium.	Cobalt.	Copper.	Dyad Iron.	Triad Iron.	Lead.	Magnesium.	Manganese.	Monad Mercury.	Dyad Mercury.	Nickel.	Potassium.	Silver.	Sodium.	Strontium.	Dyad Tin.	Tetrad Tin.	Zinc.
Oxide . .	*A & I*	*W*	*A*	*W*	*a*	*a*	*W—A*	*A & I*	*A*	*A*	*a*	*A*	*A*	*A*	*a*	*A*	*A*	*A*	*W*	*A*	*W*	*w*	*a*	*A & I*	*A*
Phosphate .	*a*	*w*	*w—a*	*a*	*a*	*a*	*A*	*a*	*a*	*a*	*a*	*a*	*a*	*a*	*a*	*a*	*a*	*a*	*w*	*a*	*W*	*a*	*a*	*a*	*a*
Silicate . .	*A—I*			*a*		*a*	*a*	*a*	*a*	*a*	*a*	*a*	*a*	*a*	*a*			*a*	*W*		*W*	*a*			*a*
Sulphate .	*W*	*W*	*a*	*I*	*w*	*W*	*W—I*	*W & A*	*W*	*W*	*W*	*W*	*A—I*	*W*	*W*	*w—a*	*w*	*W*	*W*	*W—A*	*W*	*I*	*w*		*W*
Sulphide .	*a*	*W*	*A*	*W*	*a*	*A*	*W—A*	*a—i*	*a*	*a*	*A*	*a*	*A*	*a*	*a*	*a*	*A*	*a*	*W*	*a*	*W*	*w*	*a*	*A*	*A*
Acetate . .	*W*	*W*		*W*	*w*	*w*	*W*	*w*	*w*	*W*	*w*	*W*	*W*	*w*	*w*	*w—a*	*w*	*w*	*W*	*w*	*W*	*w*	*w*	*w*	*W*
Citrate . .	*w*	*w*		*a*		*a*	*w—a*	*w*	*w*	*w*	*w*	*W*	*a*	*w*	*a*	*a*	*w—a*	*w*	*w*	*a*	*w*	*a*			*w—a*
Cyanide . .		*w*		*w—a*		*a*	*w*	*a*	*a—i*	*a*	*a—i*		*a*	*w*	*a*		*W*	*a—i*	*W*	*i*	*w*	*w*			*a*
Ferricyanide		*w*					*w*		*i*		*I*	*w*	*w—a*	*w*	*i*			*i*	*W*	*i*	*w*		*i*		*a*
Ferrocyanide		*w*		*w—a*			*w*		*i*	*i*	*i*	*I*	*a*	*w*	*a*			*i*	*W*	*i*	*w*	*w*	*i*	*i*	*a—i*
Oxalate . .	*a*	*W*	*a*	*a*	*a*	*a*	*a*	*w—a*	*a*	*a*	*a*	*a*	*a*	*a*	*w—a*	*a*	*a*	*a*	*W*	*a*	*w*	*a*	*a*	*w*	*a*
Tartrate .	*w*	*w*	*a*	*a*	*a*	*w—a*	*a*	*w*	*w*	*w*	*w—a*	*W*	*a*	*w—a*	*w—a*	*w—a*	*a*	*a*	*W*	*a*	*w*	*a*	*a*		*a*

ADDITIONS TO TABLE OF SOLUBILITY.

Aluminium and ammonium, sulphate. *W.*

Antimony, oxide. Soluble in hydrochloric acid, insoluble in nitric acid.

Antimony, sulphide. Dissolved by first converting it into oxide by heating with nitric acid, and then adding hydrochloric acid.

Antimony and potassium, tartrate. *W.*

Bismuth, basic chloride. *A.*

Bismuth, basic nitrate. *A.*

Cobalt, sulphide. Rather soluble in nitric acid, decomposed with great difficulty by hydrochloric acid.

Copper, basic acetate. Partially soluble in water, completely soluble in acids.

Copper, sulphide. Decomposed with difficulty by hydrochloric acid, readily by nitric acid.

Lead, red oxide. Converted by hydrochloric acid into chloride, by nitric acid into nitrate and insoluble binoxide.

Manganese, peroxide. Readily soluble in hydrochloric acid, insoluble in nitric acid.

Mercuric sulphide. Insoluble in hydrochloric and nitric acids separately, soluble in aqua regia.

Nickel, sulphide. Rather soluble in nitric acid, decomposed with great difficulty by hydrochloric acid.

Potassium, acid carbonate. *W.*

Potassium, bichromate. *W.*

Potassium, acid oxalate. *W.*

Potassium, acid sulphate. *W.*

Potassium, acid tartrate. *W.*

Potassium and sodium, tartrate. *W.*

Sodium, biborate. *W.*

Sodium, acid carbonate. *W.*

Sodium and ammonium, phosphate. *W.*

Stannous and stannic sulphides. Dissolved by warm hydrochloric acid, converted by nitric acid into insoluble stannic oxide. Sublimed stannic sulphide is only dissolved by warm aqua regia.

Zinc, sulphide. Readily soluble in nitric acid, rather difficultly soluble in hydrochloric acid.

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THE END.

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