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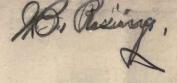
Chemistry.

Inorganic and Organic.

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BY

J. T. HEWITT, M.A., D.Sc., Ph.D., F.C.S., Professor of Chemistry;

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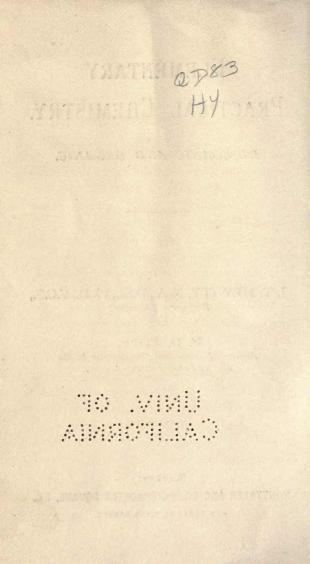
F. G. POPE,

Assistant Lecturer and Demonstrator in the People's Palace Technical Schools.

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PREFACE.

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THIS small work on qualitative analysis is intended to supply a text-book for students working according to the syllabus of the elementary stage of Practical Inorganic and Organic Chemistry. The substances for which tests are given are those contained in the new syllabus (1894), but it is hoped that the methods given for their detection and separation are such that the student will have nothing to unlearn when he proceeds to a more advanced course of qualitative analysis.

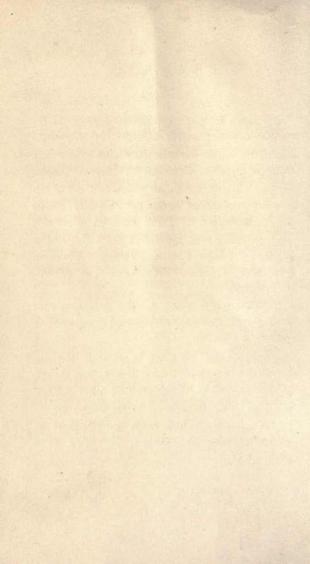
Directions are given in the making up of reagents for students who are working without the aid of a teacher.

Objection may be taken by some to the way in which it is recommended that results should be stated, but the authors find after several years practical experience in laboratory teaching, that this method leads to clearness of expression, and the rapid examination of a student's work.

> J. T. H. F. G. P.

Chemical Laboratory, People's Palace, E.

237571



HINTS FOR LABORATORY WORK.

It is necessary that all apparatus should be kept scrupulously clean, as good results cannot be expected if dirty apparatus is used.

Reagent bottles should be replaced on the shelves directly after use, and the stoppers should on no account be mixed.

Write down the results observed as soon as an experiment is made : it is hard to remember all the work done when an analysis is finished. Do not leave out an account of an experiment because it has yielded a negative result.

Before systematic analysis is begun, "preliminary tests should be made.

Distilled water should always be used—both for making up reagents and for analytical purposes.

Silver and platinum residues should not be thrown away, but put into separate stock-bottles for recovery.

Students should practise using small quantities of substance and reagents, as much time is wasted in having large quantities to filter.

Equations should be given wherever possible, to illustrate the reactions that occur.

For the method of stating the results of an analysis, see the sample analyses given at the end of the analytical tables.

REACTIONS OF THE METALS.

Lead—Dry reaction.—Heated on charcoal in the reducing flame, lead compounds give a bead of the metal. This is malleable, and marks paper.

Wet reactions.—Use a solution of lead nitrate Pb $(NO_3)_2$.

Hydrochloric acid gives a white precipitate (PbCl₂).

 $Pb(NO_3)_2 + 2HCl = PbCl_2 + 2HNO_3$.

The precipitate is soluble in boiling water, and crystallizes out again on cooling.

Hydrogen sulphide gives a black precipitate of lead sulphide (PbS).

 $Pb(NO_3)_2 + H_2S = PbS + 2HNO_3$.

In presence of much HCl the precipitate may be red. If so, dilute and pass more gas.

Sulphuric acid precipitates white lead sulphate $(PbSO_4)$.

 $Pb(NO_3)_2 + H_2SO_4 = PbSO_4 + 2HNO_3.$

Potassium chromate gives a yellow precipitate of yellow lead chromate ($PbCrO_4$).

 $Pb(NO_3)_2 + K_2CrO_4 = PbCrO_4 + 2KNO_3.$

Potassium iodide precipitates yellow lead iodide (PbI₂).

 $Pb(NO_3)_2 + 2KI = PbI_2 + 2KNO_3$.

The precipitate dissolves in much boiling water, and separates as yellow scales on cooling.

Mercury.—The salts of mercury are of two sorts : mercurous salts, corresponding to mercurous oxide Hg_2O ; and mercuric salts, corresponding to mercuric oxide HgO. The wet reactions of these two series of salts differ.

Dry reactions.—Mercury salts volatilize when heated on charcoal in the blowpipe flame.

Heated with sodium carbonate in a dry tube, a mirror of metallic mercury is formed.

Wet reactions—Mercurous salts.—Use a solution of mercurous nitrate (HgNO₃).

Hydrochloric acid gives a white precipitate of mercurous chloride (calomel).

 $HgNO_3 + HCl = HgCl + HNO_3$.

The precipitate is insoluble in boiling water, and turned black by ammonia.

 $_{2}$ HgCl + $_{2}$ NH₃ = NH₄Cl + NH₂Hg₂Cl.

Mercuric salts.—Use a solution of mercuric chloride (HgCl₂; corrosive sublimate).

[*Note.*—Mercuric chloride, unlike mercurous chloride, is soluble in water; hence mercuric salts are not precipitated by addition of hydrochloric acid.]

Hydrogen Sulphide gives a grayish ppt., passing through yellow to black mercuric sulphide (HgS).

 $HgCl_2 + H_2S = HgS + 2HCl.$

This precipitate does not dissolve in hot dilute nitric acid; but if boiled with HCl and Bromine-water, a solution of HgCl₂ is obtained.

Copper turnings become coated with a white shining deposit of metallic mercury.

Potassium iodide gives a red precipitate of mercuric iodide (HgI₂).

 $HgCl_2 + 2KI = HgI_2 + 2KCl.$

This precipitate is soluble in excess of potassium iodide.

$$HgI_2 + 2KI = K_2HgI_4.$$

Soda and potash precipitate yellow mercuric oxide.

 $HgCl_2 + 2KOH = HgO + H_2O + 2KCl.$

Ammonia gives a white precipitate.

 $HgCl_2 + 2NH_3 = NH_2HgCl + NH_4Cl.$

Stannous chloride gives a precipitate—at first white (HgCl), then turning gray (Hg).

 $2 \operatorname{HgCl}_{2} + \operatorname{SnCl}_{2} = 2 \operatorname{HgCl} + \operatorname{SnCl}_{4}.$ $2 \operatorname{HgCl} + \operatorname{SnCl}_{2} = 2 \operatorname{Hg} + \operatorname{SnCl}_{4}.$

Aluminium—Dry reaction.—Aluminium compounds heated on charcoal in the oxidizing flame yield a white mass of the oxide. If this is moistened with cobalt nitrate solution $(Co(NO_3)_2)$ and again heated, a blue mass is produced. Wet reactions.—Use a solution of potash alum, K_2SO_4 . $Al_2(SO_4)_3$. $24H_2O$.

Ammonia gives a white gelatinous precipitate of aluminium hydroxide, Al(OH)₃, insoluble in excess.

 $Al_{2}(SO_{4})_{3} + 6NH_{4}OH = 2Al(OH)_{3} + 3(NH_{4})_{2}SO_{4}$

Soda and potash give a similar precipitate, soluble in excess.

 $Al(OH)_3 + 3NaOH = Al(ONa)_3 + 3H_2O.$

The alkaline solution gives a precipitate of Al(OH)₃ on boiling with ammonium chloride.

 $Al(ONa)_3 + 3NH_4Cl = Al(OH)_3 + 3NaCl + 3NH_3.$

Calcium — Dry Reaction. — Calcium salts moistened with hydrochloric acid impart an orange colour to the Bunsen flame.

Wet reactions.—Use a solution of calcium chloride, $CaCl_2$.

Ammonium Carbonate gives a white precipitate of calcium carbonate, CaCO₃.

 $CaCl_2 + (NH_4)_2CO_3 = CaCO_3 + 2NH_4Cl.$

Ammonium Oxalate precipitates white calcium oxalate, CaC_2O_4 .

 $CaCl_{2} + (NH_{4})_{2}C_{2}O_{4} = CaC_{2}O_{4} + 2NH_{4}Cl.$

Sulphuric acid gives a precipitate of calcium sulphate, $CaSO_4$, in strong solutions, if, however the solution be dilute, the precipitate will not

appear, as calcium sulphate is slightly soluble in water.

 $CaCl_2 + H_2SO_4 = CaSO_4 + 2HCl.$

Magnesium—Dry reaction.—Heated before the blowpipe, magnesium salts leave a white residue of magnesium oxide, MgO. If this is treated with a drop of cobalt nitrate solution and again heated in the oxidizing flame a pink mass is obtained.

Wet reactions.—Use a solution of magnesium sulphate, $MgSO_4$.

The solution will give precipitates with ammonium hydroxide and carbonate if ammonium chloride is not present. If, however, ammonium chloride be present, the double chloride of ammonium and magnesium $(2NH_4Cl, MgCl_2)$ is formed, which gives no precipitate with these reagents.

[N.B.—In this way calcium and magnesium may be separated. Mix solutions of calcium and magnesium chlorides, add ammonium chloride, and then ammonia and ammonium carbonate. The white precipitate will consist of calcium carbonate, the magnesium remains in the solution.]

Caustic potash and *soda* produce white precipitates of magnesium hydroxide, $Mg(OH)_2$.

 $MgSO_4 + 2KOH = Mg(OH)_2 + K_2SO_4.$

The precipitate is soluble in ammonium chloride; magnesium hydroxide will not be

thrown down if ammonium chloride is present in the original solution.

Sodium phosphate added to a solution of a magnesium salt which contains ammonium chloride and ammonia gives a white crystalline precipitate of magnesium ammonium phosphate.

$MgSO_4 + Na_2HPO_4 + NH_4OH = Mg(NH_4)PO_4 + Na_2SO_4 + H_2O.$

Potassium—Dry reaction.—Potassium salts colour the Bunsen flame violet. Lead salts communicate a blueish violet colour to the flame, hence, if lead salts are present, potassium must be looked for in the residue left on ignition, and not in the original mixture (see Table F).

Wet reactions.—Use a solution of potassium chloride, KCl.

Platinum chloride gives a yellow crystalline precipitate of potassium chlorplatinate, K_2PtCl_a (the solution must not be too dilute).

$$2$$
KCl + PtCl₄ = K₂PtCl₆.

Fluosilicic acid gives a white gelatinous precipitate of potassium fluosilicate, K_2SiF_6 .

 $_2$ KCl + H_2 SiF₆ = K_2 SiF₆ + $_2$ HCl.

Tartaric acid precipitates acid potassium tartrate from a fairly concentrated solution of a potassium salt.

 $KCl + H_2$, $C_4H_4O_6 = KH$, $C_4H_4O_6 + HCl$.

dilute acid.	be made in	
soluble in water or	the substance should	
be	fo	
The substance given for examination will be soluble in water or dilute acid.	Before doing the wet reactions, an examination of the substance should be made in	y.
qns	doin	wa.
The	Before	the dry way.

Contraction of the second second	INFERENCE.	A salt of Hg or NH ₄ . NH ₄ . Hg.	Hg.	Pb. A sulphate.	$ \begin{array}{l} (a) \ Pb. & (b) \ A \ nitrate. \\ (c) \ Al, \ Ca, \ or \ Mg. \\ Mg. \end{array} $
	OBSERVATION.	 (a) White sublimate. (b) Smell of ammonia. (c) Metallic mirror. 	Metallic mirror.	(a) A metallic bead, which is malle- able and marks paper. (b) Place the residue on a silver coin, and moisten with water; a black stain on the silver.	$ \begin{array}{c} (a) \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \$
me and may.	EXPERIMENT.	r. Heat some of the substance in a bulb tube or dry test tube.	2. Mix with dry Na ₂ CO ₃ , and heat in a bulb tube.	$_{3}^{3}$. Mix the substance with dry $N_{a}^{3}CO_{3}^{4}$ and heat on charcoal in the reducing flame.	4. (1) Heat on charcoal at the point of the oxidizing flame. (2) Moisten the white residue with $Co(NO_{3})_{2}$, and again heat in the oxidizing flame.

INFERENCE.	K. Ca.		INFERENCE.	₽.H.A.	A chlcride. A nitrate. Oxide or sulphate.	A carbonate.
OBSERVATION.	Flame is coloured. (a) Violet. (b) Orange.	should also be made :—	OBSERVATION.	NH ₃ given off.	 (a) Pungent white fumes given off. (b) Slightly brownish fumes, having strong smell. (c) No gas given off. 	Cårbon dioxide evolved, which turns lime-water milky.
Experiment.	5. Heat some of the substance moist- ened with a little HCl on wire in the Bunsen flame.	The following preliminary tests should also be made :	EXPERIMENT.	r. Warm some of the substance with NaOH.	2. Heat with concentrated H_2SO_4 .	3. Add dilute HCl.

REACTIONS OF THE METALS. 13

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----Dissolve the substance in water if nossible--if not in dilute hydrochloric acid

Dresove the substance in water it possible—it not, in diute hydrochlora catd, and regard any residue as a precipitate of lead or mercurous chloride (Table B). When a clear solution has been obtained, add dilute hydrochloric acid to the well-cooled solution; and if a precipitate forms, filter and wash the precipitate with cold water, adding the washings to the filtrate.	White ppt. indi- Pass excess of H_3S through the filtrate, or add a considerable excess of a solution of H_2S ; warm the presence gently, and filter. [*] Wash the precipitate on the filter.	Black ppt., if Boil off excess of H_2S ; then add a few drops of HNO ₃ ; then boil, ard add Pb t or Hg(ic)salts NH ₄ Cl, NH ₄ OH in excess, and a little (NH ₄) ₂ S. Filter off precipitate, and wash on the filter.	A white ppt. in- dicates Al. To the filtrate add excess of (NHA) ₂ CO ₄ . If a precipi- tate forms, filter it off, and wash on the filter.	White ppt., if Ca Divide solution into two parts.	1. Add a solu- 2. Evaporate other tion of Na ₂ HPO ₄ . portion to dryness, Warn very gently, ignite residue to and stir. White expel amnonium ppt. Mg. Violet flame Ex.	• The solution must not be too acid, or the precipitation of the metals by H_sS will be hindered. \uparrow Lead is found in the precipitate formed by H_sS as well as in the precipitate of chlorides, as $PbCI_s$ is slightly soluble in water. For the further separation of the precipitates obtained, and the confirmation of the metals, see the fol-
Dissorve une su as a precipitate o add dilute hydro precipitate with o	White ppt. indi- cates the presence			The second		* The solution mu in the precipitate for For the further

lowing tables.

ELEMENTARY PRACTICAL CHEMISTRY. 14

TABLE B.

The precipitate obtained by the addition of HCl may contain PbCl₂ and HgCl (Group I. ppt.). These chlorides may be separated by treatment with boiling water; the PbCl₂ dissolves, and the solution is filtered from the insoluble HgCl.

The insoluble residue on the filter consists of HgCl. Confirm its composition by adding ammonia, when it will turn black from formation of NH₂Hg₂Cl. Divide hot filtrate into three parts.

(a) To one part add K_2CrO_4 . An immediate yellow ppt. consists of PbCrO₄.

(δ) Add KI to another portion, and allow to cool. PbI₂ separates in glistening yellow scales on cooling.

(c) Cool and add H_2SO_4 ; a white ppt, of $PbSO_4$ is formed.

TABLE C.

The black ppt. obtained by the action of H_2S may be tested for Pb and Hg(ic) in^{*} the following way. It is boiled with dilute HNO_i , which dissolves the PbS, forming $Pb(NO_3)_2$; whilst HgS is not dissolved by this treatment. Filter and wash.

To confirm the presence of Hg in the residue, dissolve it in HCl, with addition of a little bromine-water; boil off the excess of Br,* and add a solution of SnCl₂. A ppt., at first white (HgCl), then turning gray (Hg), indicates the presence of Hg. Evaporate the filtrate to a small bulk; cool, and add a few drops of dilute sulphuric acid. A white ppt. of $PbSO_4$ indicates the presence of Pb. Confirm the presence of Pb by heating some of the original substance on charcoal in the reducing flame—a malleable metallic bead should be obtained.

* The excess of bromine must be carefully removed, or the stannous chloride will be oxidized at the expense of the Br, and not by the HgCl₂; hence a ppt. of HgCl and Hg will not be obtained.

TABLE D.

The ppt. obtained by addition of NH_4Cl , NH_4OH , and $(NH_4)_2S$, consists of $Al(OH)_3$. Note that it is the ammonia which effects the precipitation, not the $(NH_4)_2S$.* To confirm the presence of Al, dissolve the ppt. in the least possible quantity of dilute HCl, add NaOH in slight excess, and then NH_4Cl , and boil. A white gelatinous ppt. of $Al(OH)_3$ indicates the presence of Al.

TABLE E.

The ppt. obtained by addition of $(NH_4)_2CO_3$ consists of CaCO₃. The calcium may be confirmed by dissolving a portion of the ppt. in HCl, adding NH₄Cl, NH₄OH, and $(NH_4)_2C_2O_4$. A white ppt. of CaC₂O₄ is produced.

Another portion of the ppt. should be moistened with HCl, and heated on wire in the Bunsen flame—the flame is coloured orange.

TABLE F.

The filtrate from the calcium ppt. contains magnesium and potassium. Divide into two parts.

To one part add $Na_{2}HPO_{4}$, warm, and allow to stand. A white cryst, ppt. (Mg(NH ₄) PO ₄) indicates Mg.	Evaporate the other por- tion to dryness; ignite to expel ammonium salts, and test the residue (if any), after moistening with HCl by the flame test. Violet flame=K.
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* A ppt. will be obtained in this group, even if Al is not present, if the metals Pb and Hg have not been completely removed by H_2S .

THE REACTIONS OF THE COMMON ACIDS.

H₂CO₃—H₂SO₄—HNO₃—HCl.

These acids may be divided into four different groups, viz.:--

- **Group I.**—The acid precipitated by barium chloride, but having its barium salt insoluble in acid solution—*Sulphuric acid*.
- **Group II.**—The acid precipitated from its neutral solution by barium chloride, the barium salt being insoluble in water—*Car*bonic acid.
- **Group III.**—The acid precipitated by silver nitrate, its silver salt being insoluble in dilute HNO₃—Hydrochloric acid.
- **Group IV.**—The acid whose salts are all soluble in water, and which, therefore, is not precipitated by any reagent—*Nitric acid*.

Reactions of Acid in Group I.

SULPHURIC ACID, H₂SO₄.

Sulphates are precipitated by barium chloride in the presence of hydrochloric acid, as insoluble barium sulphate.

Tests.—1. Dry sulphates heated with solid Na_2CO_3 on charcoal in the reducing flame are converted into sodium sulphide, which when

placed on a silver coin and moistened with water, gives a black stain (*i.e.*, the silver is converted into black silver sulphide, Ag₂S).

2. Soluble sulphates give with-

(A) Barium chloride or nitrate, a white precipitate of barium sulphate (BaSO₄) insoluble in hydrochloric acid, nitric acid, and ammonia.

 $BaCl_2 + H_2SO_4 = BaSO_4 + 2HCl.$

(B) Lead acetate gives a white precipitate of lead sulphate, soluble in ammonium acetate.

 $Pb(C_2H_3O_2)_2 + H_2SO_4 = PbSO_4 + 2HC_2H_3O_2.$

Reactions of Acid in Group II.

CARBONIC ACID, H₂CO₃.

Carbonates are precipitated by barium chloride in a neutral solution as insoluble barium carbonate.

 $Na_2CO_3 + BaCl_2 = BaCO_3 + 2NaCl.$

Tests.—1. The addition of acids (e.g., HCl, HNO_3) gives an evolution of carbonic acid gas (CO_2) , which on passing through lime-water turns it milky, owing to formation of calcium carbonate.

 $\mathrm{CO}_2 + \mathrm{CaH}_2\mathrm{O}_2 = \mathrm{CaCO}_3 + \mathrm{H}_2\mathrm{O}.$

2. Barium chloride gives in neutral solutions a white precipitate of barium carbonate, soluble in dilute acids with effervescence.

Reactions of Acid in Group III.

HYDROCHLORIC ACID, HCl.

Chlorides are precipitated by silver nitrate as insoluble silver chloride, e.g.—

$NaCl + AgNO_3 = AgCl + NaNO_3$.

Tests.—1. Dry chlorides on treating with strong sulphuric acid evolve hydrochloric acid gas, which turns blue litmus red, and gives dense white fumes of ammonium chloride in the presence of strong ammonia.

2. Dry chlorides heated with sulphuric acid and potassium bichromate evolve chlorine, which can be recognised by its smell, and by its bleaching of *moist* red litmus paper.

3. Solutions of chlorides give with silver nitrate a white precipitate of silver chloride, soluble in ammonia, and reprecipitated on the addition of excess of nitric acid.

4. Solutions of chlorides give with mercurous nitrate a white precipitate of calomel, which is turned black on the addition of ammonia.

Reactions of Acid in Group IV.

NITRIC ACID, HNO3.

This acid is not precipitated by any reagent, as all its salts are soluble.

Tests.—1. Solid nitrates evolve oxygen when heated, and some evolve brown nitrous fumes as well. The oxygen may be tested with a glowing splinter.

2. Solid nitrates heated with strong sulphuric acid, an equal bulk of water, and a piece of copper, give nitric oxide, turning brown on exposure to the air.

$$_{3}Cu + 8HNO_{3} = _{3}Cu(NO_{3})_{2} + _{2}NO + _{4}H_{2}O.$$

 $_{2}NO + O = N_{2}O_{3}.$

3. Add to the dry substance some strong sulphuric acid and mix well. Cool and add carefully a cold solution of ferrous sulphate. If nitric acid be present, a black ring is formed at the junction of the two liquids.

[The H_2SO_4 first liberates HNO₃. Then the nitric acid is reduced by some of the ferrous sulphate to nitric oxide, which cannot escape except by passing through the excess of ferrous sulphate; in this it dissolves, forming the black compound.

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

TABLE G.

DETECTION OF ACIDS.

Test separate portions of the original substance for chlorides, nitrates, sulphates, and carbonates, as follows :—

- (A) To the original substance add dilute HNO₃. Effervescence without smell, with a white precipitate formed on passing the gas into lime-water, indicates a carbonate.
- (B) To the original substance add strong H_2SO_4 , cool and add a solution of $FeSO_4$, a black

ring at the junction of the two liquids indicates a nitrate. Confirm by warming the original substance with sulphuric acid and a strip of metallic copper. Brown fumes evolved.

- (C) Dissolve the original substance in HCl (filter if a precipitate is formed), and add $BaCl_2$. A white precipitate insoluble in HCl and HNO₃ indicates a sulphate. Confirm by dry test.
- (D) Dissolve the original substance in HNO₃, and add AgNO₃. A white precipitate soluble in NH₄OH indicates a chloride. Confirm by the preliminary tests.

Reactions of Organic Compounds.

METHYL ALCOHOL, CH₃OH.

1. Heat with soda lime and it is converted into formic acid.

2. Distil over zinc-dust and it yields CO and H.

$$CH_3OH = CO + 2H_2$$
.

3. Heat with sodium acetate and strong sulphuric acid and it yields methyl acetate.

 $C H_3 O H + C H_3 C O O N a + H_2 S O_4 =$ CH_3COOCH_3 + NaHSO_4 + H_2O.

ETHYL ALCOHOL, C₂H₅OH.

1. Heat with $K_2Cr_2O_7$ and H_2SO_4 and aldehyde is evolved.

 $C_2H_5OH + O = CH_3CHO + H_2O.$

2. Add KHO, a strong solution of I in KI, till faint brown colour is produced, and then a drop or two of KHO till colour is destroyed, boil, and a yellow ppt. of iodoform (CHI_s) falls.

3. Heat with sodium acetate and strong sulphuric acid it yields ethyl acetate.

$C_{2}H_{5}OH + CH_{3}COONa + H_{2}SO_{4} = CH_{3}COOC_{2}H_{5} + H_{2}O + H_{2}SO_{4}.$

GLYCERIN, C₃H₅(OH)₃.

I. Evaporate to small bulk and heat with dry KHSO₄, and acrolein is evolved, recognised by its sharp and penetrating smell.

 $C_{3}H_{8}O_{3} = C_{3}H_{4}O + 2H_{2}O.$

FORMIC ACID, HCOOH.

I. Solid formates heated with strong H_2SO_4 evolve CO, which burns with a blue flame, forming CO₂. No charring takes place.

 $HCOOH = H_2O + CO.$

2. To a solution of a formate add $FeCl_3$, a deep brown red colour is formed, and, on boiling, a reddish ppt. of basic ferric formate is formed.

3. AgNO₃ in strong neutral solutions gives a white ppt. of silver formate, which is decomposed on warming, with the deposition of metallic silver on the tube.

 $AgNO_3 + NaCHO_2 = AgCHO_2 + NaNO_3.$ $2AgCHO_2 = HCO_2H + 2Ag + CO_2.$ 4. HgCl₂ gives a white ppt. of HgCl, which on heating is decomposed, and Hg is deposited as a black powder.

 $HCOOH + 2HgCl_2 = 2HgCl + 2HCl + CO_2$.

 $HCOOH + 2HgCl = 2Hg + 2HCl + CO_2$.

5. A solution of $KMnO_4$ is at once decolorized by a formate.

ACETIC ACID, CH₃CO₂H.

1. Solid acetates heated with H_2SO_4 give off acetic acid, recognised by its smell.

2. Solid acetates heated with strong H_2SO_4 and a drop or two of absolute alcohol give off ethyl acetate.

 $C H_3 C O O N a + C_2 H_5 O H + H_2 S O_4 =$ CH_3COOC_2H_5 + NaHSO_4 + H_2O.

3. FeCl₃ in strong solutions gives a red coloration, which, on boiling, forms a deep brown red ppt. of basic ferric acetate.

[The normal acetate, $Fe(C_2H_3O_2)_s$, is first formed, and this is decomposed on boiling by the excess of water, thus :—

 $Fe(C_2H_3O_2)_3 + 2H_2O = Fe(OH)_2C_2H_3O_2 + 2HC_2H_3O_2.]$

4. AgNO₃ gives a white ppt. of $AgC_2H_3O_2$, not decomposed on boiling.

 $AgNO_3 + C_2H_3O_2Na = AgC_2H_3O_2 + NaNO_3.$

OXALIC ACID COOH.

ĊOOH.

1. Solid oxalates on heating with strong

 H_2SO_4 evolve CO and CO₂, and do not char.

$$\begin{array}{c} \text{COOH} \\ \text{COOH} \end{array} = \text{CO} + \text{CO}_2 + \text{H}_2\text{O}.$$

2. $CaCl_2$ in neutral solutions gives a white ppt. of CaC_2O_4 , readily soluble in HCl.

 $Na_2C_2O_4 + CaCl_2 = 2NaCl + CaC_2O_4.$

3. AgNO₃ gives a white ppt. of $Ag_2C_2O_4$, soluble in acids and ammonia.

 $Na_2C_2O_4 + 2AgNO_3 = 2NaNO_3 + Ag_2C_2O_4.$

TARTARIC ACID, $CH(OH)CO_2H$. $CH(OH)CO_2H$.

1. Solid tartrates, on heating in a tube, blacken and give off a smell of burnt sugar.

2. Solid tartrates, heated with strong H_2SO_4 , blacken and evolve CO and CO_2 .

3. CaCl₂ in neutral solutions gives a white ppt. of CaC₄H₄O₆, soluble in HCl; also in *cold* strong KHO, but repptd. on boiling.

 $Na_2C_4H_4O_6 + CaCl_2 = 2NaCl + CaC_4H_4O_6.$

4. AgNO₃ in neutral solutions gives a white ppt. of $Ag_2C_4H_4O_6$. If this ppt. be washed and dissolved in the least possible quantity of NH₄OH and the solution warmed, Ag will be deposited on the tube.

 $2 \operatorname{AgNO}_3 + \operatorname{Na}_2 C_4 H_4 O_6 = 2 \operatorname{NaNO}_3 + \operatorname{Ag}_2 C_4 H_4 O_6.$

5. KCl in presence of a little alcohol gives a white ppt. of $KHC_4H_4O_6$.

 $\mathrm{KCl} + \mathrm{C}_{4}\mathrm{H}_{4}\mathrm{O}_{6} = \mathrm{KHC}_{4}\mathrm{H}_{4}\mathrm{O}_{6} + \mathrm{HCl}.$

REACTIONS OF THE COMMON ACIDS. 25

6. Tartrates with a strongly alkaline solution of $KMnO_4$ give a pink colouration, which on boiling forms a brown ppt.

CITRIC ACID, CH₂. COOH. COH. COOH. CH₂. COOH.

1. Solid citrates on heating blacken and give off unpleasant fumes.

2. Solid citrates on heating with strong H_2SO_4 , evolve CO, then blacken, evolving CO_2 , acetone, &c.

3. $CaCl_2$ in neutral solutions gives a white ppt. of $Ca_3(C_6H_5O_7)_2$ on boiling.

 $3CaCl_2 + 2Na_3C_6H_5O_7 = 6NaCl + Ca_3(C_6H_5O_7)_2$. No ppt. is formed in the cold.

4. AgNO₃ gives a white ppt. of $Ag_3C_6H_5O_7$ soluble in NH₄OH, but it is *not* reduced on heating.

3AgNO₃ + Na₃C₆H₅O₇ = Ag₃C₆H₅O₇ + 3NaNO₃.

5. Citrates boiled with a strongly alkaline solution of $KMnO_4$ give a green coloration, but *no* ppt.

SALICYLIC ACID, C₆H₄(OH)COOH.

1. Solid salicylates heated with CaO yield phenol, recognised by smell.

 $C_6H_4OHCOOH + CaO = C_6H_5OH + CaCO_3$.

2. FeCl₃ gives a deep purple coloration, even in very dilute solutions.

3. Bromine-water gives a white ppt.

Before proceeding with the systematic analysis, the following preliminary tests should be made :

EXPERIMENT.	OBSERVATION.	INFERENCE.
I. Heat a small portion of the sub- stance in a small dry tube.	and gives odour of burnt sugar.	trate or citrate. Presence of an oxalatę.
2. Heat a small portion of sub- stance in a dry tube with strong H_2SO_4 .	after long heating. (B) Rapid effer- vescence with char- ring, and CO, CO ₂ , and ultimately SO ₂ ⁺ given off. (C) CO [‡] only ev- olved; no charring. (D) CO and CO ₂ evolved; no char- ring.	trates and citrates. Presence of a tar- trate or citrate. Presence of a formate. Presence of an oxalate. Presence of an

* Salicylic acid is decomposed on rapid heating, thus :- $C_6H_4(OH)CO_2H = C_6H_5OH + CO_2$.

† The carbon present acts on the sulphuric acid, reducing it, thus : $C + 2H_2SO_4 = CO_2 + 2H_2O + 2SO_2$.

I Formic acid is decomposed thus :— HCO₂H=CO+H₂O. ∥ Oxalic acid is decomposed thus :—

 $H_2C_2O_4 = H_2O + CO + CO_2.$

If the substance given is a liquid, it should be tested for methyl and ethyl alcohols and glycerine in the following manner :—

(A) Methyl and ethyl alcohols.—Should the mixture smell of alcohol, make it alkaline with NaOH and distil a large portion, collect the distillate and redistil it over half its bulk of freshly ignited K_2CO_3 . Repeat the operation and take first portion of distillate only. Now note the boiling point of the liquid.

Methyl alcohol boils at 67°C and ethyl alcohol at 78°C.

The liquid should give a smell of aldehyde when heated with dilute H_2SO_4 and $K_2Cr_2O_7$.

(B) *Glycerine.*—Evaporate liquid to small bulk on water bath. A thick viscid liquid remains, which on heating with dry KHSO₄ gives pungent smell of acrolein.

$C_{3}H_{5}(OH)_{3} = C_{3}H_{4}O + 2H_{2}O.$

PREPARATION OF SOLUTION TO BE TESTED FOR ORGANIC ACIDS.

I. Dissolve small portion of substance in a little water—if possible. If solution is neutral and does not contain heavy metals (*i.e.*, gives no ppt. on treating portions with HCl, H_2S and Na_2CO_3) it can be used at once for the detection of the acids.

II. If substance does not dissolve in water or if it contains heavy metals, dissolve it in HNO₃, add H₂S, and if a ppt. is formed pass H₂S in excess and filter, boil filtrate to expel H₂S and add solid Na₂CO₃ till very alkaline, boil, and if necessary, filter. To solution add litmus and slight excess HNO₃, boil off CO₂, add ammonia till slightly alkaline and boil till excess of ammonia is expelled. Solution should now be neutral.

[H_2S is added to ppt., AS and Sb, and Na₂CO₃ to remove all remaining metals, except those of Group V.]

	dd a strong solution y.	Solution.	Boil filtrate for some time, replacing the evapored H_2O . If a ppt, forms, filter,	Solution.	To solution add FeCl ₃ . A purple coloration indicates salicylic acid (confirm by preliminary tests). A dark brown coloration, and a brown ppt. on boiling, indicates formic or acetic acid.	
EH.	IE ORGANIC ACIDS.	from heavy metals, a 2. Filter, if necessary	SoLu	Boil filtrate for some time, repl ated H_2O . If a ppt. forms, filter,	PRECIPITATE.	The ppt. consists of calcium citrate. Ppt. is soluble in HCI.
TABLE H. SEPARATION OF THE ORGANIC ACIDS.	To a strong, cold, neutral solution, free from heavy metals, add a strong solution of CaCl ₃ , and allow to stand for some time. Filter, if necessary.	PRECIPITATE.	The ppt. may contain calcium oxalate or tartrate, both. Wash and treat with cold KHO, free from ${}_{0}CO_{3^{\circ}}$ Filter, if necessary.	SOLUTION.	The filtrate may con- tain calcium tartrate. Boil filtrate ; a white ppt. confirms tartaric acid. Confirm also by AgNO ₃ test.	
		To a strong, cold, of CaCl ₂ , and allow t	PRECI	The ppt. may contain calcium oxalate or tartrate, or both. Wash and treat with cold KHO, free from K_2CO_3 . Filter, if necessary.	PRECIPITATE.	The ppt. consists of calcium oxalate. Dissolve in HCl, and add NH4,0H in excess ; a white ppt. insoluble in acetic acid, confirms ox- alic acid.

TO TEST FOR FORMIC ACID.

- (A) In absence of tartaric or citric acids.—To solution add strong AgNO₃ solution. A white ppt. is formed which immediately blackens on heating the solution, owing to reduction of AgNO₃ to Ag.
- (B) In presence of tartaric and citric acids.— Distil a portion of the substance with dilute H_2SO_4 , stopping the distillation before blackening occurs.* Neutralize distillate and test as above.

TO TEST FOR ACETIC ACID.

- (A) In absence of formic acid.—To solution add FeCl₃—a brown coloration, giving a dark brown ppt. on boiling, indicates acetic acid.
- (B) In presence of formic acid and alcohol.—If alcohol be present, boil it off. Add dilute H_2SO_4 and distil off the acetic and formic acids, boil distillate for some minutes with an equal bulk of a solution of $K_2Cr_2O_7$, and dilute H_2SO_4 in a flask with reflux tube.⁺ Distil, neutralize distillate, cool and add FeCl₃ —brown coloration and brown ppt. on boiling indicates acetic acid.

* If distillation is carried too far SO_2 might be evolved, and a black ppt. would be formed with AgNO₃.

+ This destroys formic acid and does not act on acetic. $H_2CO_2+O=CO_2+H_2O.$

Typical Analyses.

Inorganic Mixtures.

(A) The substance is a white powder.

PRELIMINARY EXAMINATION FOR METALS AND ACIDS.

EXPERIMENT.	OBSERVATION.	INFERENCE.
I. Heat some of substance in bulb tube.	A white sublimate.	Presence of an ammonium salt.
2. Mix with dry Na_2CO_3 , and heat in bulb tube.	No metallic mirror.	Absence of a mer- cury salt.
3. Mix substance with dry Na_2CO_3 , and heat on charcoal in reducing flame.	A malleable me- tallic bead formed.	Presence of a lead salt.
4. Heat on char- coal in oxidizing flame.	 (a) Yellow incrustation. (b) Substance deflagrates. 	Presence of a lead salt. Presence of a ni- trate.
5. Heat substance moistened with HCl in Bunsen flame.	A bluish - violet flame coloration.	Presence of a po- tassium or lead salt.
6. Warm some of substance in a tube with NaOH solu- tion.	$\rm NH_3$ given off.	Presence of an ammonium salt.
7. Heat some of substance in a tube with strong H_2SO_4 .	Brownish fumes evolved.	Presence of a ni- trate.
8. Add dilute HCl to original substance.	No effervescence.	Absence of a car- bonate.

Substance is easily soluble in hot water. Cool thoroughly, and add HCl in cess.	l add HCl in	and then NH4Cl,				No ppt.; ab- sence of Mg, hite.	No residue ; absence of K.
	ioroughly, and	Substance is easily soluble in hot water. Cool thoroughly, and add HCl in xcess. A white ppt. Filter, and add Filter , boil off $\operatorname{H}_{3}S$, add a few drops of HNO_{3} , and then $\operatorname{NH}_{4}\operatorname{Cl}$, need.	ormed. Presence of Pb A black ppt. NH OH in excess, and a little (NH4) ₂ S. And A black ppt. Thesence of Pb formed; pre-	03.	No ppt. ; ab- Add Na2HPO4.	No ppt.; ab- sence of Mg.	
	ter. Cool th			ss, and a little (N	Add (NH ₄) ₂ C(No ppt. ; ab-	10 000
Systematic Analysis.	ole in hot wa			No ppt. ; ab-			
1.1	is easily solub	Filter, and add H ₂ S in excess.	A black me	formed ; pre-	Hg".		
	Substance i excess.	A white ppt. formed.	Dracance of Dh	or Hg'.	a Statistics		

REACTIONS OF THE COMMON ACIDS. 31

EXAMINATION OF GROUP I. PPT.

EXPERIMENT.	OBSERVATION.	INFERENCE.
Wash ppt. with cold H_2O , and boil with H_2O .	Ppt. completely dissolves.	Presence of Pb.
Divide solution into three parts :— To (1) add K_2CrO_4 . ,, (2) ,, KI. ,, (3) Cool, and add dilute H_2SO_4 .	A yellow ppt. A yellow ppt. A white ppt.	Presence of Pb. Presence of Pb. Presence of Pb.

EXAMINATION OF GROUP II. PPT.

EXPERIMENT.	OBSERVATION.	INFERENCE.
Boil ppt. with di- lute HNO ₃ .	Ppt. completely dissolves.	Presence of lead ; absence of Hg.
Evaporate solution to small bulk, cool, and add dilute H_2SO_4 .	A white ppt.	Presence of Pb.

TEST ORIGINAL SUBSTANCE FOR AMMONIA.

EXPERIMENT.	OBSERVATION.	INFERENCE
Boil with NaOH.	Ammonia gas evolved; recognised by smell and by giving dense white fumes (of NH ₄ Cl), when a rod moist- ened with strong HCl is brought near.	Presence of am- monium.

REACTIONS OF THE COMMON ACIDS.

TEST FOR ACID.

EXPERIMENT.	OBSERVATION.	INFERENCE.
To substance add strong H_2SO_4 ; cool, and add FeSO ₄ solution.		Presence of a ni- trate.
Boil substance with sulphuric acid and a strip of cop- per.	Brown fumes evolved.	Presence of a ni- trate.

Substance contains, therefore, lead, ammonium, and nitric acid; and is, therefore, probably a mixture of lead and ammonium nitrates.

(B) The substance is a white powder.

PRELIMINARY EXAMINATION FOR METALS

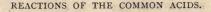
AND ACIDS.

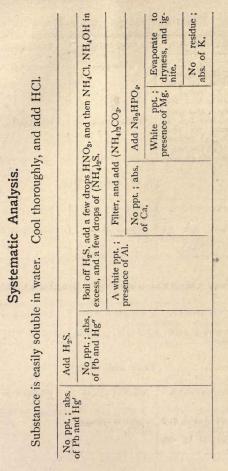
EXPERIMENT.	OBSERVATION.	INFERENCE.
I. Heat sub- stance in dry tube.	No sublimate. No smell of am- monia. No metallic mir- ror.	Abs. of mercury or ammonium salts. Abs. of an am- monium salt. Abs. of a mercury salt.
2. Heat with dry Na ₂ CO ₃ .	No metallic mir- ror.	Abs. of a mercury salt.

С

PRELIMINARY EXAMINATION FOR METALS AND ACIDS—continued.

EXPERIMENT.	OBSERVATION.	INFERENCE.
3. Mix substance with dry Na ₂ CO ₃ ; heat on charcoal in reducing flame.	 (a) No metallic bead. (b) Place residue on a silver coin, and moisten with H₂O. A black stain on the silver. 	Abs. of a lead salt. Presence of a sul- phate.
4. Heat on char- coal in oxidizing flame.	No incrustation ; white shining resi- due.	Abs. of a lead salt; presence of an aluminium, cal- cium, or magnesium salt.
Moisten white residue with $Co(NO_3)_2$, and heat again.	No distinct co- loration (<i>i.e.</i> , nei- ther quite pink nor quite blue).	(?) Presence of al- uminium and mag- nesium,
5. Heat substance moistened with HCl in Bunsen flame.	No flame colora- tion.	Abs. of potassium and calcium salts.
6. Warm sub- stance with NaOH solution.	No NH_3 evolved.	Abs. of ammon- ium salts.
7. Heat substance with strong H_2SO_4 .	No white fumes evolved. No brown fumes evolved.	Abs. of chlorides. Abs. of nitrates.
8. Add dilute HCl to original sub- stance.	No effervescence.	Abs. of a carbon- ate.





EXAMINATION OF GROUP III. PPT.

EXPERIMENT.	OBSERVATION.	INFERENCE.
Dissolve ppt. in very small quantity of dilute HCl, add slight excess of NaOH, and then NH ₄ Cl, and boil.	A white ppt.	Presence of Al.
Confirm ppt. by heating on char- coal, and moisten- ing with $Co(NO_3)_2$.	A blue mass.	Confirms Al.

TO CONFIRM Mg.

EXPERIMENT.	OBSERVATION.	INFERENCE.
Heat ppt. on charcoal moistened with $Co(NO_3)_2$.	A pink mass.	Presence of Mg.

TEST ORIGINAL SUBSTANCE FOR AMMONIUM.

EXPERIMENT.	OBSERVATION.	INFERENCE.
Boil with NaOH.	No NH ₃ evolved.	Abs. of NH ₄ .

TEST FOR ACID.

EXPERIMENT.	OBSERVATION.	INFERENCE.
Dissolve sub- stance in HCl, and add BaCl ₂ . Confirm by dry test.	A white ppt., in- soluble in HCl and HNO ₃ .	Presence of H_2SO_4 .

REACTIONS OF THE COMMON ACIDS. 37

Substance contains aluminium and magnesium and sulphuric acid, and is therefore probably a mixture of aluminium and magnesium sulphates.

Organic Mixtures.

(A) The substance is a white powder.

PRELIMINARY EXAMINATION.

EXPERIMENT.	OBSERVATION.	INFERENCE.
Heat substance in a dry tube.	Immediate black- ening and smell of burnt sugar.	Presence of a tar- trate or citrate.
Heat substance with strong H_2SO_4 .	Rapid efferves- cence, with charring and evolution of CO, CO_2 , and ulti- mately SO_2 .	Presence of a tar- trate or citrate.

Systematic Analysis.

EXPERIMENT.	OBSERVATION.	INFERENCE.
To a strong and exactly neutral solu- tion, free from heavy metals, add CaCl ₂ , and allow to stand for ten minutes.	No ppt.	Absence of oxal- ates and tartrates.
Boil solution for five minutes.	White ppt.	Presence of a ci- trate.
Filter, and add $FeCl_3$.	Dark brown co- loration and dark brown ppt. on boil- ing.	Presence of a for- mate or acetate.

EXAMINATION OF PPT. PRODUCED BY CaCl₂.

EXPERIMENT.	OBSERVATION.	INFERENCE.
Boil ppt. with HCl. Confirm by reactions, page 25.	It dissolves.	Presence of a ci- trate.

TO CONFIRM FORMIC OR ACETIC ACID.

EXPERIMENT.	OBSERVATION.	INFERENCE.
Distil portion of original substance with dilute H ₂ SO ₄ , stopping distillation before blackening occurs; and to dis- tillate add AgNO ₃ .		Presence of a for- mate.

Subtance was therefore a mixture of a formate and a citrate.

(B) The substance is a colourless liquid.

EXPERIMENT.	OBSERVATION.	INFERENCE.
Heat substance in a dry tube.	Liquid boils; the vapour has an al- coholic smell.	Presence of me- thyl or ethyl alco- hol.
Heat residual liquid with strong H_2SO_4 .	A very irritating odour of acrolein.	Presence of gly- cerine.
Test reaction of original solution.	Neutral.	No free organic acid.
Burn a drop or two of liquid on Pt foil.	No residue left.	Absence of salts of organic acids.

REACTIONS OF THE COMMON ACIDS. 39

Distil a portion of the original liquid, collect the distillate, redistil it over half its bulk of freshly ignited K_2CO_3 , repeat the process and take the first portion of the distillate. Determine its boiling point—found to be 78°. The substance is ethyl alcohol.

Test for glycerine in the residual liquid left after distilling off the alcohol, by evaporating with KHSO₄ and heating strongly. A pungent odour of acrolein indicates glycerine.

The substance is therefore a mixture of glycerine and ethyl alcohol.

Preparation of Reagents.

In making up reagents for use in the laboratory, distilled water must be always used.

Hydrochloric acid.— The strong pure acid should be diluted with three times its volume of water.

Nitric acid.—The strong pure acid should be diluted with three times its volume of water.

Sulphuric acid.—The strong pure acid should be diluted with four times its volume of water, allowed to stand overnight to allow the lead sulphate to settle, and then siphoned off. [Care must be taken to pour the strong acid into the water, and NOT the water into the acid.]

Sodium hydrate.—Dissolve one part by weight of the solid in ten times its weight of water.

Ammonium hydrate.—Dilute '880 ammonia with three times its volume of water.

Lime-water.—Shake up one part of slaked lime with about one hundred times its weight of water, allow to stand overnight and decant the clear liquid. Preserve in a tightly stoppered bottle.

Ammonium chloride.—Dissolve one part by weight of the solid in eight times its weight of water.

Ammonium carbonate.—Dissolve one part by weight of the solid in five times its weight of water, and add its own weight of '880 ammonia.

Barium chloride.—Dissolve one part by weight of the solid in ten times its weight of water.

Calcium chloride.-Make a saturated solution.

Cobalt nitrate.—Dissolve one part by weight of the solid in ten times its weight of water.

Ferrous sulphate.—Dissolve one part by weight of the solid in ten times its weight of *cold* water, and add a few drops of dilute sulphuric acid.

Ferric chloride.—Dissolve one part by weight of the solid in ten times its weight of water.

Potassium iodide.—Dissolve one part by weight of the solid in twenty times its weight of water.

Potassium chromate.—Dissolve one part by weight of the solid in twenty times its weight of water.

Sodium phosphate. — Dissolve one part by weight of the solid in ten times its weight of water.

REACTIONS OF THE COMMON ACLES, 41.

Stannous chloride. — Dissolve or e part by weight of the solid in its own weight of strong hydrochloric acid, and dilute with four times its volume of water. A small piece of granulated tin should always be kept in the solution.

Silver nitrate.—Dissolve one part by weight of the solid in twenty times its weight of water.

For organic analysis dissolve one part by weight of the solid in five times its weight of water.

Platinum chloride. — Dissolve one part by weight of the solid in ten times its weight of water.

Ammonium oxalate.—Dissolve one part by weight of the solid in twenty-four times its weight of water.

Sulphuretted hydrogen. — Make a saturated solution of the gas. The gas should first be washed through water to free it from any small quantities of acid present.

Bromine-water.—Add about five grammes of bromine to one hundred grammes of water, and shake well.

Ammonium sulphide.—Add to three volumes of the strong solution twenty volumes of water and two volumes of '880 ammonia.

Lead acetate.—Dissolve one part by weight of the solid in ten times its weight of water, and add a little acetic acid.

Mercuric chloride.—Dissolve one part by weight of the solid in sixteen times its weight of water.

Mercury (ous) Ammonium. Aluminium. Magnesium. Mercury (ic) Potassium. Calcium, Sodium .ead. Acetate (w) w w w (w) w W w w Carbonate w a a a a a w w Citrate w w a a w a 3. W W Formate w W w w w w W w Chloride w w w (w) w a w w w Nitrate w W W w w w w w w Oxalate a W a a a a a w W Salicylate w w w w Sulphate (w) w (w) w w w w w Tartrate w a a a a a w w

TABLE OF SOLUBILITY OF SALTS.

w signifies that the compound is soluble in water.

(w) signifies that the compound is difficultly soluble in water. a signifies that the compound is soluble in acids.

i signifies that the compound is insoluble in water and acids.

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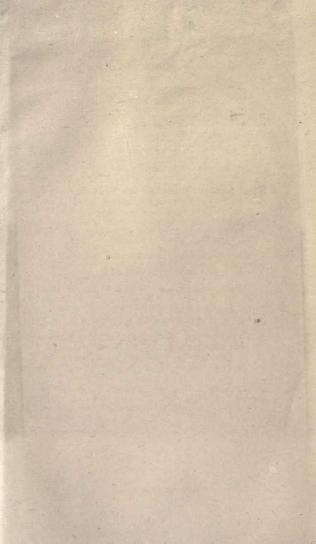
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