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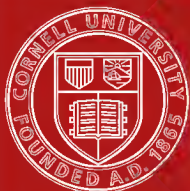
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SHORT COURSE

IN

G. C. CALDWELL

QUALITATIVE

CHEMICAL ANALYSIS.

BY

JOHN H. APPLETON, A. M.,

PROFESSOR OF CHEMISTRY IN BROWN UNIVERSITY.

THIRD EDITION.

PHILADELPHIA  
COWPERTHWAIT & CO.

1878.

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1878.

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## PREFACE TO THE THIRD EDITION.

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THE author, having used this little work for several years in his own classes, offers a new and revised edition to his fellow-teachers.

The features of the book that are considered of especial importance are—

First, its brevity and compactness;

Second, the direct and simple course of analysis prescribed;

Third, the large number of formulas and reactions presented.

The experienced teacher will at once appreciate the importance of these characteristics.

There are many valuable works on this subject, but most of them are too bulky and too costly; so, also, some of the best of them give processes of analysis that are far too complex and involved, for a beginner. As to formulas and reactions,—these are unquestionably of great importance: no matter whether the student is hoping to become an expert professional analyst, or merely to gain mental discipline, he must master reactions and formulas if he would really comprehend the subject.

The especial aim of the author is to afford a short but instructive course in this fascinating branch of Chemistry.

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# QUALITATIVE ANALYSIS.

## CHAPTER I.

### FIRST GROUP OF METALS.

#### *Description.*

1. THE first group includes those metals which are precipitated upon addition of Chlorohydric acid (H Cl) to their solution.

The metals are—

Lead,	Pb	(Plumbum),	usually II. (sometimes IV.).
Silver,	Ag	(Argentum),	“ I.
Mercury, (when it is in its mercurous form),	Hg <sub>2</sub>	(Hydrargyrum),	II.

#### *Types of Compounds.*

2. Plumbic oxide,	Pb O,	Pb = O.
Plumbic di-oxide,	Pb O <sub>2</sub> ,	Pb { = O. = O.
Argentive chloride,	Ag Cl,	Ag — Cl.
Mercurous chloride,	Hg <sub>2</sub> Cl <sub>2</sub>	Cl — Hg — Hg — Cl.

Mercuric compounds belong to the Second group. For if we add Chlorohydric acid to a Mercuric compound, Mercuric chloride is formed; but Mercuric chloride is *soluble*, hence it does not appear as a precipitate in the First group, but passes on to the next group, where it *is* precipitated.

### The Process.

3. To the solution to be tested, add a few drops of dilute Chlorohydric acid. If a precipitate appears, thoroughly agitate the solution, and then allow the precipitate to subside. To the clear liquid add a few more drops of the acid. If no precipitate appears, it shows that enough acid has been used; but if more precipitate appears, repeat the agitation, etc. just described. It is important to add enough acid, but a large excess must be carefully avoided.

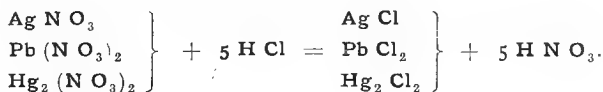
4. The precipitate indicates the probable presence of some member or members of the First group.

Filter; *the filtrate* may contain soluble chlorides of metals of later groups; *the precipitate* contains the insoluble chlorides of the metals of the First group. It may contain the following compounds:

Plumbic chloride,  $\text{Pb Cl}_2$ , crystalline; white.

Argentichloride,  $\text{Ag Cl}$ , flocculent; white, changing to purple or black upon exposure to sunlight.

Mercurous chloride,  $\text{Hg}_2 \text{Cl}_2$ , flocculent; white.



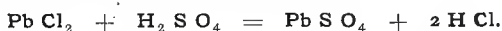
5. Remove the precipitate and boil it in water.

Filter. Boil the precipitate in a new portion of water, and again filter. Hot water dissolves the Chloride of lead, but does not dissolve the Chloride of silver nor the Mercurous chloride. (It is important to dissolve the whole of the Plumbic chloride, even if several repeated boilings are necessary.)

*The Filtrate containing Lead.*

6. Divide this solution into three portions. (Sometimes crystals of  $\text{PbCl}_2$  form, as the solution cools.)

7. **First Portion.**—To this, add *dilute* Sulphuric acid ( $\text{H}_2\text{SO}_4 + \text{Aq.}$ ) If Lead is present, a white precipitate of Plumbic sulphate ( $\text{PbSO}_4$ ) appears.

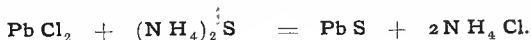


Common *concentrated* Sulphuric acid usually holds, dissolved in itself, some Plumbic sulphate. This Plumbic sulphate may be precipitated by diluting the strong acid; by taking the *clear* acid from which the  $\text{Pb S O}_4$  has subsided, we have a dilute acid that is free from this impurity.

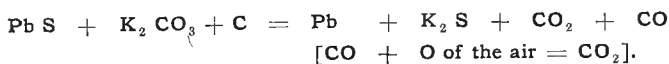
8. **Second Portion.**—To this, add Potassic di-chromate ( $\text{K}_2\text{Cr}_2\text{O}_7$ ). If Lead is present, a yellow precipitate of Plumbic chromate ( $\text{PbCrO}_4$ ), also called chrome-yellow, appears.



9. **Third Portion.**—To this, add Ammonic sulphide. If Lead is present, a brown or black precipitate appears.



The black precipitate is Plumbic sulphide ( $\text{Pb S}$ ). Dry this precipitate, and then fuse it (together with adhering filter-paper, if necessary) on charcoal, with a small amount of Potassic carbonate. Small, bright, metallic globules of Lead may be obtained. It is important to allow them to cool, and then to try their malleability. Lead is very malleable. Bismuth is brittle; so also is antimony.



The brown precipitate, above referred to, is a compound containing, in irregular proportions, both  $\text{Pb Cl}_2$  and  $\text{Pb S}$ ; it changes entirely to black,  $\text{Pb S}$ , upon addition of a sufficient quantity of Ammonic sulphide.

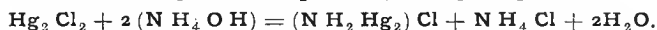
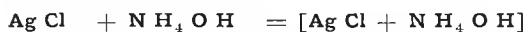
The solution called Ammonic sulphide may contain :

Ammonic sulphide,	$(\text{N H}_4)_2 \text{S}$ .
Ammonic di-sulphide,	$(\text{N H}_4)_2 \text{S}_2$ .
Ammonic sulphhydrate,	$(\text{N H}_4) \text{S H}$ .
Ammonic hydrate,	$(\text{N H}_4) \text{O H}$ .

### *The Precipitate Containing Silver and Mercury.*

**10.** (Sometimes this precipitate, which was originally white, is found to change, *on the surface*, to a purplish or black color. This change is due to the influence of the sunlight, and points to probable presence of Silver.)

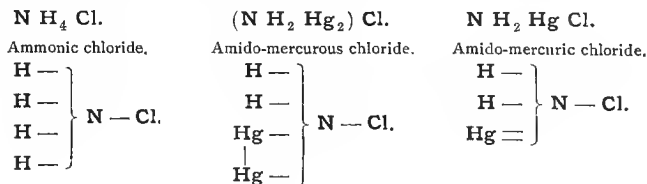
Transfer the precipitate to a test-tube, and add Ammonic hydrate ( $\text{NH}_4\text{OH}$ ). It is well to agitate the mixture and to warm it gently, but not to boil it. The reagent *dissolves the AgCl*, but it changes the  $\text{Hg}_2\text{Cl}_2$  into a black substance called Amido-mercurous chloride or Black-precipitate ( $\text{NH}_2\text{Hg}_2\text{Cl}$ ).



There is also an Amido-mercuric chloride. It is white, and its formula is  $\text{N H}_2 \text{Hg Cl}$ . It is produced by adding Ammonic hydrate to Mercuric solutions. It is also called White-precipitate.

### *Diagrams,*

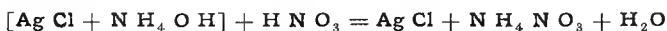
to show relationship of Black-precipitate and White-precipitate, to Ammonic chloride.



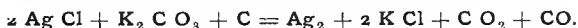


### *The Test for Silver.*

**11.** Filter; test the *filtrate* for Silver as follows. Add carefully Nitric acid ( $\text{HNO}_3$ ) until the solution has a slightly acid reaction to litmus. A white precipitate or a milkiness indicates the presence of Silver. The white substance is Argentic chloride. It re-appears because its solvent ( $\text{NH}_4\text{OH}$ ) has been neutralized by the  $\text{HNO}_3$  added.



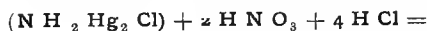
If the amount of  $\text{AgCl}$  is sufficient, it should be fused on charcoal, as described in paragraphs 9 and 13.



It frequently occurs that the quantity of  $\text{Ag Cl}$  produced by the experiment just described is very small. Unless great care is used, the principal portion of  $\text{Ag Cl}$  is not dissolved by Ammonia, but remains mixed with the Black-precipitate. (See paragraph 13, NOTE.)

### *The Test for Mercury.*

**12.** Test the Black-precipitate for Mercury as follows. Dissolve it in the smallest possible quantity of *Aqua-regia* (a mixture of two measures of  $\text{HCl}$  and one measure of  $\text{HNO}_3$ ). This gives rise to Mercuric chloride ( $\text{HgCl}_2$ ).

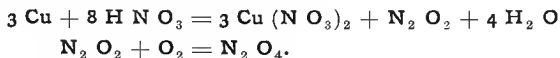


**13.** Evaporate the solution to the bulk of a few drops, and then dilute with water and filter.

NOTE.—Frequently an insoluble residue of  $\text{Ag Cl}$  appears at this stage. It is that portion which was not dissolved by the treatment with Ammonia, described in paragraph 10. Dry this precipitate, and then fuse it on charcoal before the blowpipe, adding a few granules of Potassic carbonate. Bright, white globules of metallic Silver are easily obtained.

**14.** To the solution containing  $\text{HgCl}_2$  add a few strips of cleaned copper wire or copper foil.

The copper is cleaned by immersing it in Nitric acid and then washing it in water. The acid removes the coating of black Cupric oxide ( $\text{Cu O}$ ) or red Cuprous oxide ( $\text{Cu}_2 \text{O}$ ), and forms Cupric nitrate,  $\text{Cu (N O}_3)_2$ , which latter salt dissolves and washes away.



**15.** If Mercury is present, the copper quickly changes color; after a few minutes, remove the wire or foil from the solution and gently wipe it dry. It acquires a bright silvery appearance because of a coating of metallic Mercury.



**16.** Place the pieces of wire or foil in a glass tube closed at one end, and heat them. The Mercury should leave the copper, and passing up in the state of vapor should condense in metallic globules in the upper part of the tube.

### *Additional Experiments.*

**17.** Fill a beaker half full of water; add *carefully* nearly as much common concentrated Sulphuric acid. Allow the whole to stand twenty-four hours.

Pour away the clear part. Add a little water to the sediment (which is  $\text{PbSO}_4$ ) and filter. Dry the filter with its precipitate. Finally, fuse the precipitate with  $\text{K}_2\text{CO}_3$  on charcoal. Globules of metallic Lead should be obtained.

**18.** Fuse, on charcoal, a small piece of metallic Lead. Observe (*a*) that a yellow coating of Plumbic oxide ( $\text{PbO}$ ) is formed on the coal; (*b*) that the metal remains fused for a

long time after the heat is withdrawn; (c) that the globule, after solidifying, is malleable.

19. Fuse, on charcoal, a small fragment of Silver.

20. Heat, on charcoal, a *very* small globule of metallic Mercury. It will boil and evaporate. (The fumes are noxious.)

### 21. Common Forms of First Group Metals.

Metallic lead,		Pb.
Litharge,	Plumbic oxide; yellow scales or powder,	Pb O.
Varieties of Red lead,	Di-plumbic tri-oxide,	Pb <sub>2</sub> O <sub>3</sub> .
	Tri-plumbic tetroxide,	Pb <sub>3</sub> O <sub>4</sub> .
	Tetra-plumbic pent-oxide,	Pb <sub>4</sub> O <sub>5</sub> .
Sugar of lead,	Plumbic acetate; brown or white,	Pb O <sub>2</sub> $\overline{A}_2$ .
White lead,	Plumbic hydro-carbonate (with oil),	Pb <sub>3</sub> H <sub>2</sub> (CO <sub>4</sub> ) <sub>2</sub> .
Nitrate of lead,	Plumbic nitrate; white crystals,	Pb (N O <sub>3</sub> ) <sub>2</sub> .
Chrome yellow,	Plumbic chromate,	Pb Cr O <sub>4</sub> .
Silver coin (or token),		Ag alloyed with Cu.
Nitrate of silver,	Argentive nitrate; white crystals,	Ag N O <sub>3</sub> .
Lunar-caustic,	(Nitrate of silver that has been fused).	
Quicksilver,	Metallic Mercury,	Hg.
Calomel,	Mercurous chloride; white powder,	Hg <sub>2</sub> Cl <sub>2</sub> .
	(See, also, Common Forms of Second Group Metals, page 28.)	



## CHAPTER II.

### SECOND GROUP OF METALS.

#### *Description.*

**22.** THE Second Group includes those metals which (*after the removal of the preceding Group*) are precipitated by Sulphydic acid ( $H_2S$ ), in presence of Chlorohydric acid ( $HCl$ ).

The metals are—

FIRST DIVISION,	{	Arsenic,	As,	III. or V.
		Antimony,	Sb,	(Stibium), III. or V.
		Tin,	Sn,	(Stannum), II. or IV.
SECOND DIVISION,	{	Gold,	Au,	(Aurum), I. or III.
		Platinum,	Pt,	II. or IV.
THIRD DIVISION,	{	Copper,	Cu, or $Cu_2$ ,	(Cuprum), II.
		Bismuth,	Bi,	III. or V.
		Mercury (when it is in its mercuric form),	Hg,	II.
		Cadmium,	Cd,	II.

#### *Types of Compounds.*

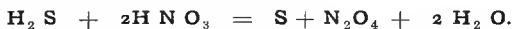
Arsenious,	As $Cl_3$ ,	As <sub>2</sub> O <sub>3</sub> .
Arsenic,	As $Cl_5$ ,	As <sub>2</sub> O <sub>5</sub> .
Antimonious,	Sb $Cl_3$ ,	Sb <sub>2</sub> O <sub>3</sub> .
Antimonic,	Sb $Cl_5$ ,	Sb <sub>2</sub> O <sub>5</sub> .
Stannous,	Sn $Cl_2$ ,	Sn O.
Stannic,	Sn $Cl_4$ ,	Sn O <sub>2</sub> .

Aurous,	Au Cl <sub>1</sub> ,	Au <sub>2</sub> O.
Auric,	Au Cl <sub>3</sub> ,	Au <sub>2</sub> O <sub>3</sub> .
Platinous,	Pt Cl <sub>2</sub> ,	Pt O.
Platinic,	Pt Cl <sub>4</sub> ,	Pt O <sub>2</sub> .
Cuprous,	Cu <sub>2</sub> Cl <sub>2</sub> ,	Cu <sub>2</sub> O.
Cupric,	Cu Cl <sub>2</sub> ,	Cu O.
Bismuthous,	Bi Cl <sub>3</sub> ,	Bi <sub>2</sub> O <sub>3</sub> .
Bismuthic,		Bi <sub>2</sub> O <sub>5</sub> .
Mercuric,	Hg Cl <sub>2</sub> ,	Hg O.
Cadmic,	Cd Cl <sub>2</sub> ,	Cd O.

NOTE.—Gold and Platinum rarely occur in solutions; they occur oftenest in alloys; their detection and recognition are omitted here, but are described under Metals and Alloys, Chapter VI.

### The Process.

**23.** The solution to be tested must be evaporated carefully to dryness, if free Nitric acid is present. This acid decomposes, and so renders useless, the Sulphuretted-hydrogen (to be hereafter added.)



The residue from evaporation must be dissolved in water and a few drops of Chlorohydric acid.

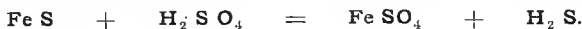
Pass into the solution, thus prepared, a current of Sulphydic gas (H<sub>2</sub>S), or add the water-solution of H<sub>2</sub>S.

A precipitate indicates the probable presence of some member or members of this Group.

Upon diluting solutions with water, a white precipitate sometimes appears. This may be due to presence of Bismuth or of Antimony, or both.



The H<sub>2</sub>S gas is usually made by addition of *dilute* Sulphuric acid to *washed* Ferrous sulphide.

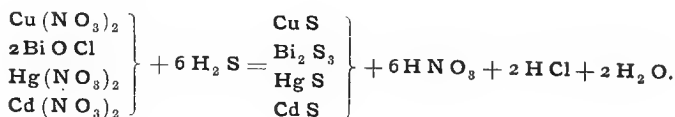
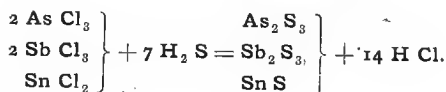


*It is essential that Sulphuretted-hydrogen be used in quantity sufficient to precipitate wholly, all the members of this Group that are present.*

**24.** Filter; *the filtrate* contains the members of succeeding Groups, and in the soluble form; *the precipitate* contains the insoluble Sulphides of the members of this Group. It may contain—

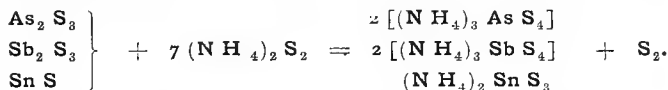
Arsenious sulphide,	$\text{As}_2 \text{S}_3$ ,	lemon yellow.
Arsenic sulphide,	$\text{As}_2 \text{S}_5$ ,	lemon yellow.
Antimonious sulphide,	$\text{Sb}_2 \text{S}_3$ ,	orange.
Antimonic sulphide,	$\text{Sb}_2 \text{S}_5$ ,	orange.
Stannous sulphide,	$\text{Sn S}$ ,	brownish-black.
Stannic sulphide,	$\text{Sn S}_2$ ,	yellow.
Aurous sulphide,	$\text{Au}_2 \text{S}$ ,	black.
Auric sulphide,	$\text{Au}_2 \text{S}_3$ ,	black.
Platinic sulphide,	$\text{Pt S}_2$ ,	black.
Cuprous sulphide,	$\text{Cu}_2 \text{S}$ ,	black.
Cupric sulphide,	$\text{Cu S}$ ,	black.
Bismuthous sulphide,	$\text{Bi}_2 \text{S}_3$ ,	black.
Mercuric sulphide,	$\text{Hg S}$ ,	white, yellow, red, brown, and finally black.
Cadmic sulphide,	$\text{Cd S}$ ,	yellow.

### *Reactions.*



25. Remove the precipitated Sulphides from the filter by adding *yellow* Ammonic di-sulphide,  $(\text{NH}_4)_2\text{S}_2$  to them; gently warm the mixture for some time.

The yellow Ammonic di-sulphide dissolves the Sulphides of arsenic, antimony and tin; it leaves the others undissolved.



*Arsenic, Antimony, and Tin, Discussed.*

26. For the purpose of explaining this dissolving action, it should be observed that the compounds of Arsenic and of Antimony resemble those of Phosphorus and of Nitrogen, while the compounds of Tin resemble those of Carbon. Thus,

*Triad Compounds of the ous Form.*

N	$\text{H}_3 \text{ N}$	$\text{N Cl}_3$	—	$\text{N}_2 \text{ O}_3$	$\text{H N O}_2$ .
P	$\text{H}_3 \text{ P}$	$\text{P Cl}_3$	$\text{P}_2 \text{ S}_3$	$\text{P}_2 \text{ O}_3$	$\left\{ \begin{array}{l} \text{H}_3 \text{ P O}_3. \\ \text{H}_3 \text{ As O}_3. \end{array} \right.$
As	$\text{H}_3 \text{ As}$	$\text{As Cl}_3$	$\text{As}_2 \text{ S}_3$	$\text{As}_2 \text{ O}_3$	
Sb	$\text{H}_3 \text{ Sb}$	$\text{Sb Cl}_3$	$\text{Sb}_2 \text{ S}_3$	$\text{Sb}_2 \text{ O}_3$	$\text{H Sb O}_2$

*Pentad Compounds of the ic Form.*

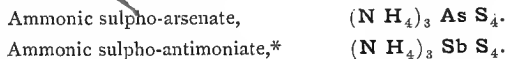
N	—	—	—	$\text{N}_2 \text{ O}_5$	$\text{H N O}_3$ .
P	—	$\text{P Cl}_5$	$\text{P}_2 \text{ S}_5$	$\text{P}_2 \text{ O}_5$	$\left\{ \begin{array}{l} \text{H}_3 \text{ P O}_4. \\ \text{H}_3 \text{ As O}_4. \end{array} \right.$
As	—	$\text{As Cl}_5$	$\text{As}_2 \text{ S}_5$	$\text{As}_2 \text{ O}_5$	
Sb	—	$\text{Sb Cl}_5$	$\text{Sb}_2 \text{ S}_5$	$\text{Sb}_2 \text{ O}_5$	$\text{H Sb O}_3$ .

27. But the correspondence is carried out still further; thus, there exist the following salts:

Ammonic [oxy] phosphite,	$(\text{N H}_4)_3 \text{ P O}_3$ .
Ammonic [oxy] arsenite,	$(\text{N H}_4)_3 \text{ As O}_3$ .
Ammonic [oxy] phosphate,	$(\text{N H}_4)_3 \text{ P O}_4$ .
Ammonic [oxy] arsenate,	$(\text{N H}_4)_3 \text{ As O}_4$ .

Now, corresponding with these salts, there are formed, by the process of

digesting in Ammonic di-sulphide, the following sulphur-salts, *which salts are soluble* :

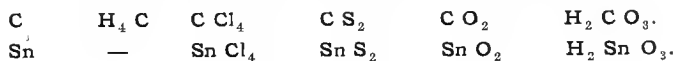


28. Again, consider the following tables :

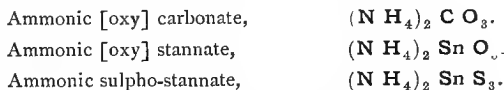
*Dyad Compounds of the dyad Form.*



*Tetrad Compounds of the tetrad Form.*



Further, the correspondence is carried to salts, thus :



These last three salts are soluble in water ; the third is the one produced by influence of the digestion in Ammonic di-sulphide.

29. The purpose of the foregoing discussion is to show that Arsenic, Antimony, and Tin tend to form acid radicles, which dissolve in alkaline solvents.

But, on the other hand, Copper, Bismuth, Mercury, and Cadmium show but little tendency to produce either anhydrides or acids or salts ; it thus becomes apparent why they do not form sulphur-salts, and so do not dissolve by the treatment now under discussion.

---

\* But the oxy-salts of Antimony correspond with the Nitrogen salts ;  
 thus to Ammonic [oxy] nitrate,  $(\text{N H}_4) \text{N O}_3$ ,  
 and Ammonic [oxy] nitrite,  $(\text{N H}_4) \text{N O}_2$ ,  
 correspond Ammonic [oxy] antimoniate,  $(\text{N H}_4) \text{Sb O}_3$ ,  
 and Ammonic [oxy] antimonite,  $(\text{N H}_4) \text{Sb O}_2$ .

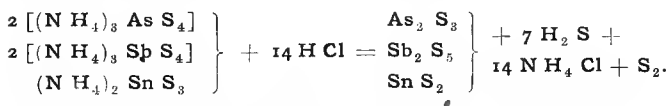


**The Process.**—(Continued.)

**30.** Filter; test the *filtrate* for Arsenic, Antimony, and Tin; test the *precipitate* for Copper, Bismuth, Mercury, and Cadmium. (It is desirable, at this stage, to wash the moist precipitate from the filter-paper into a small beaker, and to allow it to remain in the beaker until convenient to test it. See paragraph 40.)

**The Filtrate Containing Arsenic, Antimony, and Tin.**

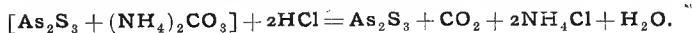
**31.** To this, add sufficient dilute Chlorohydric acid to produce distinct acid reaction. This stronger acid, decomposes the *sulphur-salts*, liberates  $H_2S$  with effervescence, and re-precipitates the metals as sulphides.



The sulphides re-appear as yellow or orange flakes. (Observe that although Tin may have been in the *stannous* form originally, it is now always changed to the stannic form, and appears as the yellow  $SnS_2$ .)

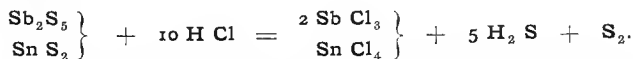
**The Test for Arsenic.**

**32.** Filter; and digest the precipitate in Ammonic carbonate,  $(NH_4)_2CO_3$ . This reagent has the power of dissolving the Sulphide of arsenic. Filter again; and test the filtrate for Arsenic by carefully adding dilute Chlorohydric acid. Effervescence of  $CO_2$  ensues, while a yellow flaky precipitate of  $As_2S_3$  appears.

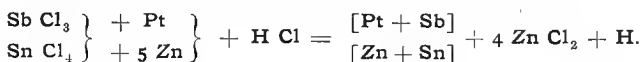


### *Antimony and Tin, Separated.*

**33.** The yellowish precipitate, consisting of  $\text{Sb}_2\text{S}_3$  and  $\text{SnS}_2$ , is now to be dissolved by boiling in concentrated Chlorohydric acid. (Perform the operation under a hood.)



**34.** Evaporate off the principal portion of the  $\text{HCl}$ , and then dilute slightly with water. Place a piece of platinum in this solution; in contact with the platinum, place a strip of zinc. During the galvanic action which ensues, a black coherent coating of metallic Antimony will form upon the platinum, while a gray spongy precipitate of metallic Tin will collect loosely upon the zinc. (Sometimes the Tin floats about in fragments in the liquid; this is frequently due to the influence of the rapidly-escaping hydrogen, which is liberated, freely, near the zinc.)



### *The Test for Antimony.*

**35.** After rinsing the platinum with water, transfer it to a casserole; drop *one or two drops* of concentrated Nitric acid upon it. (The *black* color suddenly disappears, owing to the formation of *white* Met-antimonic acid ( $\text{HSbO}_3$ ); but the Antimony is not dissolved.)



(Addition of *much* Nitric acid must be avoided, since it interferes with subsequent precipitation.) To dissolve the Met-antimonic acid, add Tartaric acid and water, and then boil; the Antimony now dissolves. To the solution, add

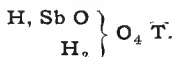
Sulphuretted-hydrogen-water; a reddish precipitate of  $\text{Sb}_2\text{S}_3$  constitutes the test for Antimony.

**36.** Tartaric acid has this formula:  $\text{H}_4\text{O}_4$  ( $\text{C}_4\text{H}_2\text{O}_2$ ),

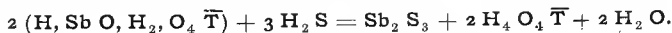
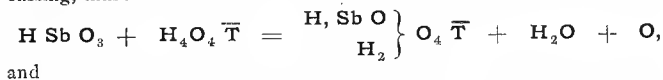
or, 
$$\begin{array}{l} \text{H}_2 = \\ \text{H}_2 = \end{array} \text{O}_4 = (\text{C}_4\text{H}_2\text{O}_2), \text{ or, abridged, } \text{H}_4 \text{O}_4 \bar{\text{T}}.$$

Two of the hydrogen atoms are replaceable by metals; two are not. Thus we may form the compounds, Potassic tartrate,  $\left. \begin{array}{l} \text{K}_2 \\ \text{H}_2 \end{array} \right\} \text{O}_4 \bar{\text{T}}$ , also Hydro-potassic tartrate,  $\left. \begin{array}{l} \text{H K} \\ \text{H}_2 \end{array} \right\} \text{O}_4 \bar{\text{T}}$ .

Further, the compound radicle Antimonyl ( $\text{SbO}$ ) may be substituted for one of the replaceable hydrogen atoms, so as to form Hydro-antimonylic tartrate,



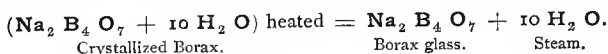
This latter is the compound formed in the experiment we are now discussing, thus:



### *The Tests for Tin.*

**37.** Gently rub the spongy metallic Tin from the zinc. (If practicable, wash it with water by decantation.) To the Tin, apply two tests.

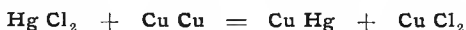
**38.** *First Test.*—Make a borax bead upon platinum wire. Color this bead very slightly, by fusing into it a minute fragment of Cupric sulphate ( $\text{CuSO}_4 + 5\text{H}_2\text{O}$ ). Into this bead, fuse (in the lower reducing flame of the Bunsen lamp) a minute fragment of the metallic Tin. A dull-red coloration of Cuprous oxide ( $\text{Cu}_2\text{O}$ ), formed by the reducing action of the Tin, constitutes this test.



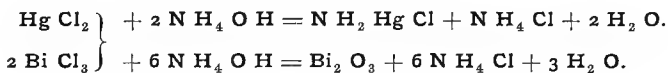


*The Tests for Mercury.*

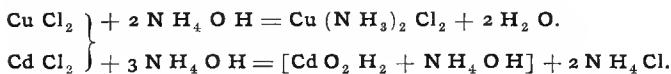
41. To *one portion*, add a fragment of clean copper wire. The Mercury, if present, will alloy itself with the copper, giving the latter a bright silvery appearance when rubbed,—if not before rubbing. Confirm this test, as described under the First Group (page 12).



42. To *the other portion* of the solution, add a slight excess of Ammonic hydrate. This reagent precipitates Mercury and Bismuth,—the former as Amido-mercuric chloride ( $\text{NH}_2\text{HgCl}$ ), also called White-precipitate,—the latter as Bismuthous oxide ( $\text{Bi}_2\text{O}_3$ ).

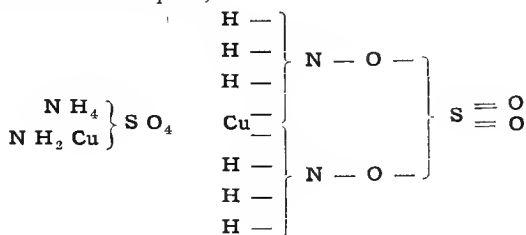


43. At the same time the Copper and Cadmium are dissolved, the former (the Copper) affording a beautiful blue color to the solution.

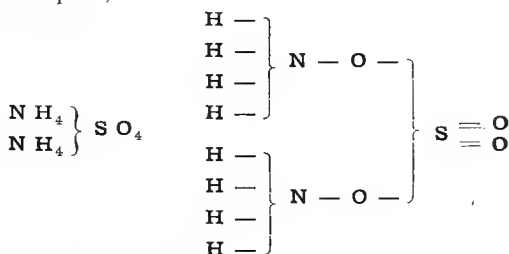


When Ammonic hydrate is added to a solution of Copper, it affords at first a light-blue precipitate of Cupric hydrate; but upon further addition of the reagent, the precipitate dissolves and affords a deep-blue solution. Such solutions may be viewed as containing ammonium salts, in which, a part of the hydrogen of the ammonium is replaced by Copper. Illustrative diagrams are given on the following page.

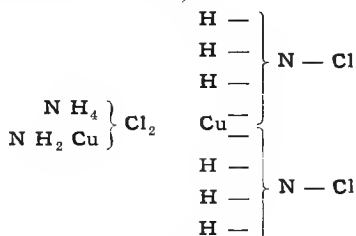
Cupric di-ammonic sulphate,



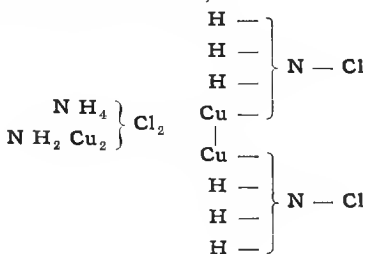
Ammonic sulphate,



Cupric di-ammonic chloride,



Cuprous di-ammonic chloride,



### *The Tests for Bismuth.*

44. Filter the mixture just discussed; dissolve the precipitate in a small amount of Chlorohydric acid. Evaporate this solution to the bulk of one or two drops; then pour these drops into a tube of cold water. A white precipitate of Bismuth oxychloride ( $\text{BiOCl}$ ) indicates the presence of Bismuth.

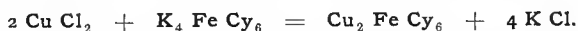
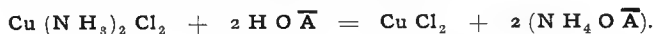


45. As an *additional test* for Bismuth, dissolve some of the  $\text{BiOCl}$  in a few drops of Chlorohydric acid. Add this to a hot solution of Plumbic iodide ( $\text{PbI}_2$ ). Cool the whole. The Plumbic iodide crystallizes, not with its proper yellow color, but with a reddish-brown color if Bismuth is present.

### *The Tests for Copper and Cadmium.*

46. To the filtrate, supposed to contain Copper and Cadmium (and which is deep-blue when much Copper is present), add sufficient Acetic acid to neutralize the Ammonia present. Then divide the solution into two parts.

47. To the *first portion*, add a few drops of Potassic ferro-cyanide; if Copper is present, a reddish-brown precipitate of Cupric ferro-cyanide appears.



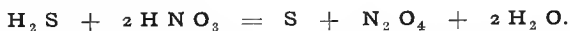
48. To the *second portion*, add Sulphuretted-hydrogen; if Cadmium alone is present, a yellow precipitate appears. If Copper is present, it gives a black sulphide, which will

conceal the yellow color of Cadmic sulphide when the latter is also present. If the precipitate is dark, filter it; then transfer the precipitate to a test-tube, and warm it with a solution of Potassic cyanide (KCy); the latter substance dissolves the Cupric sulphide. If Cadmium is present, its yellow sulphide (CdS) now becomes visible.

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### *Additional Experiments.*

**49.** Pass some Sulphuretted-hydrogen gas into some concentrated Nitric acid in a test-tube; (*a*) The solution becomes warm; (*b*) Brown fumes of  $N_2O_4$  are evolved; (*c*) Sulphur is liberated, and appears as a fine milkiness or as a gummy mass.



**50.** Place in the bottom of a tube, closed at one end, a fragment of Arsenic *of the size of the head of a pin*. Heat the tube *very gently*; observe (*a*) the smell, (*b*) the mirror or crystals of metallic Arsenic, (*c*) crystals of  $As_2O_3$  farther up the tube.

**51.** Place in the bottom of a tube closed at one end, a fragment of White-arsenic ( $As_2O_3$ ) *of the size of the head of a pin*. Heat the tube *very gently*, and observe the crystals formed in the upper part of the tube as the vapor cools; they are right octahedrons.

**52.** Place in the bottom of a tube closed at one end, a fragment of White-arsenic of the size of the head of a pin. Above it, place a fragment of charcoal which has been previously heated to redness in another tube. Now heat the White-arsenic and charcoal; the latter will *reduce* the former to metallic Arsenic, which will appear as a shining, black mirror farther up the tube.



**53.** *Reinsch's Test.*—Take a fragment of White-arsenic of the size of the head of a pin; add to it a few drops of pure HCl and about 100 c.c. of water. Allow some cleaned copper wires to remain half-an-hour in the moderately-warm solution, so prepared. The copper becomes covered with an iron-gray coating, which is an Arsenide of copper. Dry the wires, by pressing between filter-paper, and then heat them in a tube, as in Experiment 50. The test is by no means conclusive of presence of Arsenic unless distinct octahedral crystals are thus obtained on the upper part of the tube.

**54.** Fuse a fragment of Antimony, on charcoal; observe its fumes, and the crystalline coating formed about the globule, upon cooling.

**55.** Fuse a small quantity of Tartar-emetic ( $K, SbO, H_2, O_4, \bar{T}$ ) on charcoal; observe that the globule of Antimony, thus obtained, is brittle.

**56.** Boil a few fragments of metallic Antimony, in Nitric acid; it is oxydized, but does not dissolve.

**57.** Fuse a fragment of metallic Tin, on charcoal.

**58.** Boil a fragment of Tin, in Nitric acid; it is oxydized, but does not dissolve.

**59.** Hold in one hand, the end of a piece of Platinum wire; in the other hand, the end of a piece of fine Copper wire. Simultaneously place the two disengaged ends of wire in a lamp-flame. Observe the difference in the conducting powers of the metals.

**60.** Endeavor to fuse a piece of Copper wire, on charcoal before the blowpipe.

**61.** Fuse, on charcoal, a fragment of metallic Bismuth.

### 62. Common Forms of Second Group Metals.

White arsenic,	Arsenious oxide,	$\text{As}_2 \text{O}_3$ .
Orpiment,	Arsenious sulphide,	$\text{As}_2 \text{S}_3$ .
Scheele's green,	Arsenite of copper, } Hydro-cupric arsenite, }	$\text{H Cu, As O}_3$ .
Arsenate of soda,	{ Hydro-di-sodic arsenate, $\text{H Na}_2 \text{As O}_4 + 12 \text{H}_2 \text{O}$ . or Sodic arsenate, $\text{Na}_3 \text{As O}_4 + 12 \text{H}_2 \text{O}$ .	
Metallic antimony,		<b>Sb.</b>
Tartar emetic,	Potassio-antimonylic tartrate,	$\text{K Sb O H}_2 \text{O}_4 (\text{C}_4 \text{H}_2 \text{O}_2)$ .
Metallic tin,	Block tin,	<b>Sn.</b>
Tin crystals,	Muriate of tin, Stannous chloride,	$\text{Sn Cl}_2 + 2 \text{H}_2 \text{O}$
Stannate of soda,	Sodic stannate,	$\text{Na}_2 \text{Sn O}_3$ .
Metallic copper,		<b>Cu.</b>
Brass,		An alloy of copper and zinc.
Red oxide of copper,	Cuprous oxide,	$\text{Cu}_2 \text{O}$ .
Black oxide of copper,	Cupric oxide,	<b>Cu O.</b>
Blue vitriol,	Cupric sulphate,	$\text{Cu S O}_4 + 5 \text{H}_2 \text{O}$ .
Metallic bismuth,		<b>Bi.</b>
Basic nitrate of bismuth,	Bismuthyl nitrate,	$(\text{Bi O}) \text{N O}_3$ .
Pearl-white,	Bismuthyl chloride,	$(\text{Bi O}) \text{Cl}$ .
Corrosive sublimate,	Mercuric chloride,	$\text{Hg Cl}_2$ .
Cinnabar, (natural) } Vermilion (artificial) }	Mercuric sulphide,	<b>Hg S.</b>
Iodide of cadmium,	Cadmic iodide,	$\text{Cd I}_2$ .

CHAPTER III.  
THIRD GROUP OF METALS.

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*Description.*

**63.** THE Third Group includes those metals which (*after the removal of the preceding Groups*) are precipitated by Ammonic sulphhydrate in presence of Ammonic hydrate.

The metals are—

FIRST DIVISION.	{	Cobalt,	Co,	II. (sometimes IV. or VI.).
		Nickel,	Ni,	II. (IV. or VI.).
SECOND DIVISION.	{	Iron,	Fe (Ferrum),	II., IV. or rarely VI.
		Manganese, Mn,		II., IV., VI. or VIII.
		Chromium, Cr,		(rarely II.), IV., VI.
THIRD DIVISION.	{	Zinc,	Zn,	II.
		Aluminum, Al,		IV.

*Types of Compounds.*

Cobaltous,		Co O.
Cobaltic,		Co <sub>2</sub> O <sub>3</sub> .
Nickelous,		Ni O.
Nickelic,		Ni <sub>2</sub> O <sub>3</sub>
Ferrous,	Fe Cl <sub>2</sub>	Fe O.
Ferric,	Fe Cl <sub>6</sub>	Fe <sub>2</sub> O <sub>3</sub> .
Ferrate,		K <sub>2</sub> Fe O <sub>4</sub> .

Manganous,	$\text{Mn Cl}_2$ .	
Manganese,		$\text{Mn O}_2$ .
Manganic,		$\text{Mn}_2 \text{O}_3$ .
Manganate,		$\text{K}_2 \text{Mn O}_4$ .
Per-manganate,		$\text{K}_2 \text{Mn}_2 \text{O}_8$ .
Chromic,	$\text{Cr}_2 \text{Cl}_6$	$\text{Cr}_2 \text{O}_3$ .
Chromate,		$\text{K}_2 \text{Cr O}_4$ .
Zinc,	$\text{Zn Cl}_2$	$\text{Zn O}$ .
Zincate,		$\text{Na}_2 \text{Zn O}_2$ .
Aluminic,	$\text{Al}_2 \text{Cl}_6$	$\text{Al}_2 \text{O}_3$ .
Aluminate,		$\text{Na}_2 \text{Al}_2 \text{O}_4$ .

### *The Process.*

**64.** To the solution to be tested (usually the filtrate from the preceding Group), add first, sufficient Ammonic hydrate to produce alkaline reaction. This neutralizes the free HCl, which always comes from the preceding Group; it also combines with any  $\text{H}_2\text{S}$  remaining in the same filtrate. (There is thus formed  $\text{NH}_4\text{SH}$ , the very reagent next to be added.) Then add Ammonic sulphhydrate in sufficient quantity to precipitate the whole of all of the metals of this Group present. (A *large* excess must be carefully avoided.)

**65.** All the metals (except Aluminum and Chromium) are precipitated as sulphides. Aluminum and Chromium appear to have little affinity for sulphur, and not to form sulphides by liquid processes,—they are precipitated as hydrates.

**66.** Filter; *the filtrate* contains the members of succeeding Groups in the soluble form.

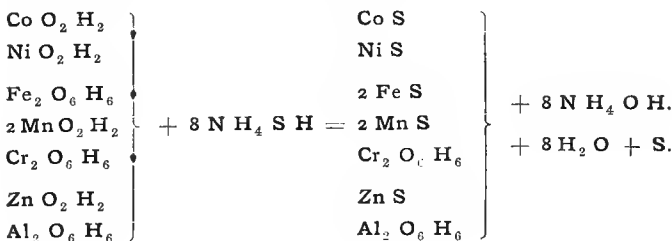
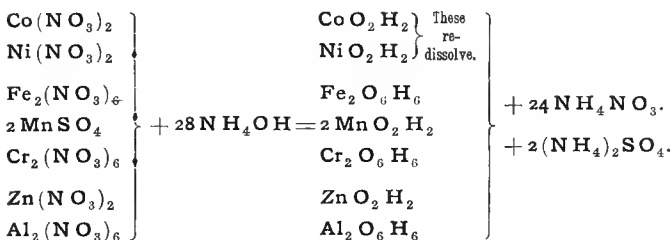
The precipitate contains the members of this Group, in the following forms :

Cobaltous sulphide,	Co S,	black.
Nickelous sulphide,	Ni S,	black.
Ferrous sulphide,	Fe S,	black.
Manganous sulphide,	Mn S,	flesh-colored.
Chromic hydrate,	Cr <sub>2</sub> O <sub>6</sub> H <sub>6</sub> ,	dull green.
Zinc sulphide,	Zn S,	white.
Aluminic hydrate,	Al <sub>2</sub> O <sub>6</sub> H <sub>6</sub> ,	white.

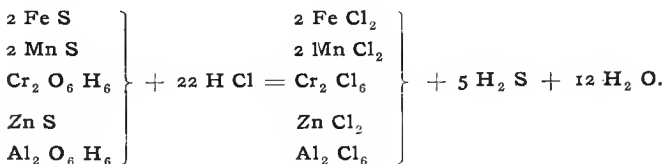
**Reactions.**



(The first set of the following reactions takes place *only* when no H<sub>2</sub> S is present in the solution.)



67. Transfer the precipitate from the filter to a casserole, add dilute pure Chlorohydric acid, and gently warm it. The Chlorohydric acid dissolves every member of the precipitate except the Cobaltous and Nickelous sulphides (CoS and NiS); these are undissolved and unchanged.



68. Filter; *the filtrate* contains Chlorides of iron, manganese, chromium, zinc, and aluminum.

*The precipitate* contains Sulphides of cobalt and of nickel.

### *The Precipitate to be Tested for Cobalt and Nickel.*

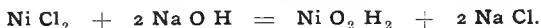
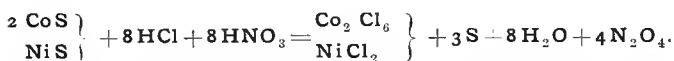
69. First, test for Cobalt as follows. Fuse some borax ( $\text{Na}_2\text{B}_4\text{O}_7 + 10\text{H}_2\text{O}$ ) in a loop in the end of a platinum wire. A clear and colorless bead must be produced. Into this bead, fuse some of the black precipitate under examination. Both sulphides are oxidized by the oxygen of the air; the sulphur is burned off and escapes; while the two oxides are dissolved by the bead, forming Sodio-cobaltous borate ( $\text{Na}_2\text{CoB}_4\text{O}_8$ ), which has a deep-blue color, and Sodio-nickelous borate ( $\text{Na}_2\text{NiB}_4\text{O}_8$ ), which is violet when hot, and brown when cold.

70. If the blue color *is obtained*, it indicates presence of Cobalt. (But Nickel *may* also be present, since the blue

color always overpowers the other.) If the blue color is *not obtained*, the bead shows whether Nickel is present or absent.



**71.** A special test for Nickel may be made as follows. Dissolve the black precipitate of CoS and NiS, in a few drops of Aqua-regia; then evaporate the solution nearly to dryness. To the residual solution add a small fragment of solid Potassic nitrite ( $\text{KNO}_2$ ), and then some Acetic acid. Allow the whole to stand for twenty-four hours. The whole of the Cobalt should now be precipitated as a yellow precipitate—a complex Potassio-cobaltic nitrite (said to be  $\text{K}_6\text{Co}_2\text{O}_{12}6(\text{N}_2\text{O}_2) + x\text{H}_2\text{O}$ ). Filter, and test the filtrate for Nickel by adding Sodid hydrate. A light-green, flaky precipitate of Nickelous hydrate indicates Nickel. (Its color becomes more distinct after twenty-four hours' standing.) The test may be confirmed by fusing a portion of the precipitate in a borax bead. The Nickel bead is violet when hot, brown when cold.

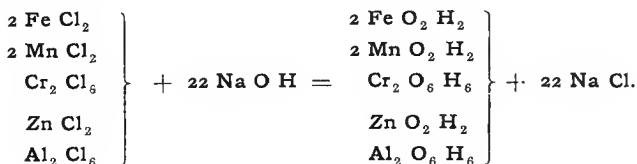


(The yellow Potassio-cobaltic nitrite also, may be tested for Cobalt, by fusing in a borax bead.)

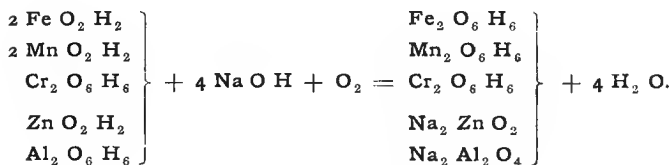
**The Filtrate to be Tested for Iron, Manganese, Chromium, Zinc, and Aluminum.**

**72.** Evaporate the solution nearly to dryness, to remove  $\text{H}_2\text{S}$  and the excess of  $\text{HCl}$ . (This is important for the success of the succeeding operations.)

**73.** Add excess of Sodic hydrate ( $\text{NaOH}$ ), and boil. An abundant precipitate of hydrates of the metals is produced, but the Hydrates of zinc and of aluminum redissolve, forming Sodic zincate ( $\text{Na}_2\text{ZnO}_2$ ) and Sodic aluminate ( $\text{Na}_2\text{Al}_2\text{O}_4$ ).



**74.** During the boiling, as above described, with excess of  $\text{NaOH}$  and with free access of air, the substances change as expressed below :



Filter; the *filtrate* contains Sodic zincate and Sodic aluminate.

The *precipitate* contains Ferric hydrate, Manganic hydrate, and Chromic hydrate. Dry the precipitate for the next test.



**The Precipitate Tested for Iron, Manganese,  
and Chromium.**

**75.** Fuse upon a platinum foil (which may be held on a short stick of charcoal) a small quantity of a mixture of Potassic carbonate ( $K_2CO_3$ ), and Potassic nitrate ( $KNO_3$ ). (The latter salt is a powerful oxydizing agent; the former serves mainly as a flux.) Now place upon the platinum foil, as above prepared, a portion of the dried precipitate.

(If it is difficult to separate the precipitate from the filter-paper, the introduction of the latter, in small portions, is not inadmissible; but it must not be forgotten that *deflagration* of considerable violence may ensue when Potassic nitrate is heated with carbonaceous matter.)

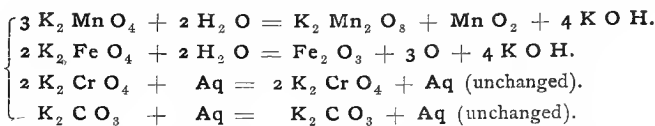
Three new salts are produced by the fusion, viz.:

Potassic ferrate,	$K_2 Fe O_4$ .
Potassic manganate,	$K_2 Mn O_4$ .
Potassic chromate,	$K_2 Cr O_4$ .

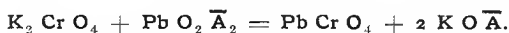
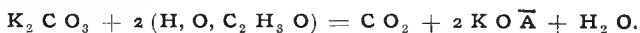


**76. For Manganese.**—The deep-green color of the Potassic manganate, constitutes the test for Manganese.

**77. For Chromium.**—Boil the fused mass in a small amount of water. A series of complex reactions takes place. In case of Manganese, Potassic per-manganate ( $K_2Mn_2O_8$ ) and Manganese di-oxide ( $MnO_2$ ) are formed. Insoluble Ferric oxide is produced. Potassic chromate dissolves unchanged, but it is always attended by some of the excess of Potassic carbonate—a fact which must not be overlooked.



78. Filter; to the filtrate add Acetic acid, and boil violently. (This is to decompose  $\text{K}_2\text{CO}_3$  and to expel  $\text{CO}_2$ . If it is not done, the subsequent step will produce a white precipitate of Plumbic carbonate, which will conceal the test sought.) Now add a few drops of Plumbic acetate. A yellow precipitate of Plumbic chromate shows presence of Chromium.

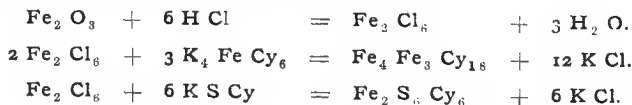


79. For Iron.—Test the precipitate obtained by the operation described in paragraph 77, as follows. Boil it in a small quantity of pure Chlorohydric acid. Dilute it with water. Divide the solution into two parts.

To one portion add a few drops of Potassic ferrocyanide ( $\text{K}_4\text{FeCy}_6$ ). If Iron is present, a blue precipitate of Ferric ferrocyanide, called Prussian blue ( $\text{Fe}_4\text{Fe}_3\text{Cy}_{18}$ ), will appear.

To the other portion, add a few drops of Potassic sulpho-cyanate ( $\text{KSCy}$ ). If Iron is present, a blood-red coloration (no precipitate) will appear.

(It must be remembered that the two reagents employed here detect Iron *when it is in the ferric form*, but not when it is in the ferrous form. Solutions of Iron in the ferrous form give a deep-blue precipitate, called Turnbull's blue, upon addition of Potassic ferricyanide.)

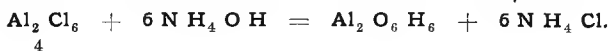
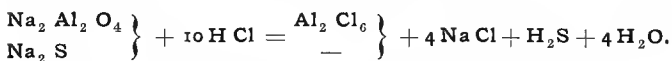
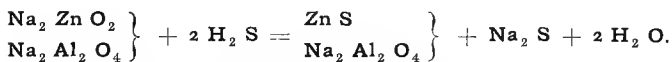


***The Filtrate to be tested for Zinc and Aluminum.***

**80. For Zinc.**—To the solution (paragraph 74), add an abundance of  $H_2S$  (sufficient to precipitate all Zinc present). A white or slaty-white precipitate of Zinc sulphide ( $ZnS$ ) constitutes the test. (Sometimes the precipitate, at this point, is nearly or quite black. This may be due to Lead, which is either an accidental impurity, or is derived from the original solution, and has failed to be arrested by the  $HCl$  used for the First Group, and by the  $H_2S$  used for the Second Group.)

If the precipitate is black, filter it; then warm it with Acetic acid. This should dissolve  $ZnS$ , and leave  $PbS$  unchanged. Filter, and to the filtrate add some Potassic hydrate ( $KOH$ ), and then  $H_2S$ . A white precipitate indicates Zinc.

**81. For Aluminum.**—To the filtrate—from the original, white, gray or black precipitate, supposed to be  $ZnS$ —add sufficient  $HCl$ , to produce acid reaction to litmus. Boil, to expel  $H_2S$ , and to collect sulphur; filter again, to get rid of precipitated sulphur. To this filtrate, add sufficient Ammonic hydrate to produce alkaline reaction; a flocculent precipitate of  $Al_2O_6H_6$  constitutes the test.



### *Additional Experiments.*

82. Make a clear bead of borax, and into it fuse a minute fragment of Ferrous sulphate; colors varying from yellow to red may be produced. The ferric compounds give lighter colors. The ferroso-ferric compounds give bottle-green colors.

83. Under similar circumstances (paragraph 82), Chromium compounds give emerald-green beads. Manganese compounds give, in the outer blowpipe flame, amethyst-colored beads, which lose their color in the inner flame, owing to a change to the Manganous form.

84. Heat a minute fragment of iron wire, on charcoal; it oxydizes, but remains un-fused (unless the finest piano-forte wire be used).

85. Heat a fragment of metallic Zinc on charcoal; it fuses at once, changing into vapor, which burns with a blue flame, and producing abundant fumes of white Zinc oxide ( $ZnO$ ); the deposited oxide, however, is yellow when hot.

86. Make a solution of Ammonio-ferrous sulphate, and divide it into three parts; to one part, add Potassic ferrocyanide—no dark-blue precipitate should appear. To the second portion, add two drops of Nitric acid and eight drops of Chlorohydric acid, and, after boiling, add Potassic ferrocyanide—a dark-blue precipitate should now appear. To the third portion, add Potassic ferricyanide—a dark-blue precipitate should appear at once.

87. Take a few drops of solution of  $K_2Cr_2O_7$ ; add a few drops of  $HCl$ , and then pass  $H_2S$  through the solution; it turns green from the reduction of  $H_2CrO_4$  to  $Cr_2Cl_6$ .

88. Warm a fragment of green Potassic manganate in water; allow the  $MnO_2$  to subside; decant the purple solution of Potassic per-manganate ( $K_2Mn_2O_8$ ), and add to it some Ethylic alcohol and dilute  $H_2SO_4$ ; the color disappears, owing to the reduction of  $K_2Mn_2O_8$  to Manganous sulphate ( $MnSO_4$ ).

### 89. Common Forms of Third Group Metals.

Nitrate of cobalt, Cobaltous nitrate; red crystals,	$Co(NO_3)_2 + 6H_2O$ .
Metallic nickel,	Ni.
Nickel coins,	{ Alloys of copper, zinc and nickel or copper and nickel.
Double sulphate of nickel and ammonia,	$(NH_4)_2SO_4 + NiSO_4 + 6H_2O$ .
Wrought iron,	Fe.
Steel, } Cast iron, }	{ Compounds of iron with varying amounts of carbon.
Copperas or green vitriol,	Ferrous sulphate, $FeSO_4 + 7H_2O$ .
Prussian blue,	Ferric ferro-cyanide, $Fe_4Fe_3Cy_{18}$ .
Pyrolusite, natural	Manganese di-oxide, $MnO_2$ .
Permanganate of potash,	Potassic permanganate, $K_2Mn_2O_8$ .
Bi-chrome,	Potassic di-chromate, $K_2Cr_2O_7$ .
Chrome yellow,	Plumbic chromate, $PbCrO_4$ .
Chrome orange,	Basic Plumbic chromate, $(PbCrO_4 + PbO)$ or $(Pb_2CrO_5)$ .
Spelter,	Metallic Zinc, Zn.
Zinc white,	Zinc oxide, ZnO.
White vitriol,	Zinc sulphate, $ZnSO_4 + 7H_2O$ .
Alum,	Ammonio-aluminic sulphate, $(NH_4)_2SO_4 + Al_2(SO_4)_3 + 24H_2O$ .
Concentrated Alum,	Aluminic sulphate, $Al_2(SO_4)_3 + 18H_2O$ .
Red liquor, solution of	Aluminic acetate, $Al_2O_6\bar{A}_6$ .

## CHAPTER IV.

### FOURTH GROUP OF METALS.

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#### *Description.*

**90.** THE Fourth Group includes those metals which (*after the removal of the preceding Groups*, and after addition of Ammonic chloride,  $\text{NH}_4\text{Cl}$ ), are precipitated by Ammonic carbonate,  $(\text{NH}_4)_2\text{CO}_3$ , in presence of Ammonic hydrate ( $\text{NH}_4\text{OH}$ ).

The metals are—

Barium,	Ba,	II.	(rarely IV.).
Strontium,	Sr,	II.	(rarely IV.).
Calcium,	Ca,	II.	(rarely IV.).

#### *Types of Compounds.*

Baric chloride,	Ba Cl <sub>2</sub>	—
Baric peroxide,	—	Ba O <sub>2</sub> .
Strontic nitrate,	Sr (N O <sub>3</sub> ) <sub>2</sub>	—
Strontic peroxide,	—	Sr O <sub>2</sub> .
Calcic sulphate,	Ca S O <sub>4</sub>	—
Calcic per-oxide,	—	Ca O <sub>2</sub> .

#### *Remarks.*

**91.** NOTE I. Certain special precautions must be observed in the treatment of the members of this Group. The separation of the metals is difficult, because—

1st. The corresponding salts of Barium, Strontium and Calcium are very similar in their properties (such as solubility in water, acids, alcohol, etc.).

2d. All the salts of these metals (with unimportant exceptions) are white, whence they cannot be distinguished by the help of color.

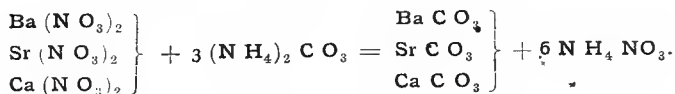
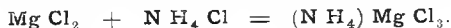
3d. These metals readily form white insoluble sulphates with that Sulphuric acid *which is present as an impurity* in most ordinary acids and reagents. The turbidity thus produced is likely to be confounded with the test sought.

NOTE II. In the course of analysis, the experimenter must aim to *have every solution perfectly clear* before applying any reagent as a test. If one filtration is not sufficient to make the solution *perfectly clear*, the filtration must be repeated until the object is attained.

NOTE III. Before proceeding to test for members of the Fourth Group, it is well to evaporate, carefully and to dryness, the filtrate from the Third Group. To the residue, add water and a few drops of pure Chlorohydric acid; then boil, filter and commence the following process.

### *The Process.*

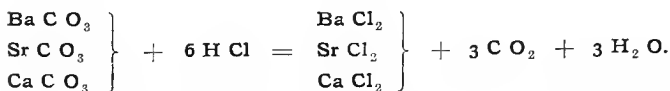
92. To the solution to be tested, add Ammonic chloride ( $\text{NH}_4\text{Cl}$ ). (It combines with Magnesium salts, and gives rise to double salts of Magnesium and Ammonium, which double salts are not precipitated by subsequent addition of Ammonic carbonate. Magnesium is thus reserved for the Fifth Group.) Next add Ammonic hydrate in quantity sufficient to make the solution alkaline to litmus. Warm the solution, and add Ammonic carbonate. This gives a white precipitate of carbonates of the metals.



93. Filter; the *filtrate* contains the members of the Fifth Group.

The *precipitate* contains the members of the Fourth Group, in the form of carbonates.

Wash the carbonates into a test-tube, and add a small amount of pure Chlorohydric acid. It will dissolve the carbonates, and change them into soluble chlorides.



**94.** Place the solution—so formed—in a casserole, and carefully evaporate to dryness. A crystalline residue is obtained; it consists of Baric chloride ( $\text{BaCl}_2$ ), Strontic chloride ( $\text{SrCl}_2$ ), Calcic chloride ( $\text{CaCl}_2$ ).

Test a *small portion* of the crystalline residue with the spectroscope, by aid of a drop of water and a *clean* platinum wire.

The *Barium* spectrum is characterized, principally, by a series of green bands between the Sodium line (Fraunhofer line D) and the line F.

The *Strontium* spectrum is recognized, principally, by a beautiful pure-blue line between F and G.

The *Calcium* spectrum is recognized by an orange-red line, to the left of the Sodium line, and by a green line at a similar distance to the right of the Sodium line.

### *The Crystalline Residue.*

**95.** To the remaining portion of the crystalline residue add Ethylic alcohol,  $(\text{C}_2\text{H}_5)\text{OH}$ . Rub the crystals, thoroughly, with the closed end of a clean test-tube. It is important that the crystals be thoroughly pulverized, in order that the Ethylic alcohol may have favorable conditions for exerting its solvent power. It dissolves the Strontic chloride and the Calcic chloride, but it leaves the Baric chloride undissolved (with the exception of a very small quantity, which is dissolved).



96. Filter the alcoholic liquid, and then—in order the more effectually to dissolve the Strontic and Calcic chlorides—treat the undissolved residue with another portion of alcohol, as before, rubbing the crystals with a test-tube. Filter this liquid, and mix its filtrate with the former one.

### *The Undissolved Portion, Tested for Barium.*

97. Test this residue as follows :

(a) Take a small portion of it upon a *clean* platinum wire, and heat it *for several minutes* in the outer mantle of the Bunsen lamp-flame. A yellowish-green color in the flame constitutes a test for Barium.

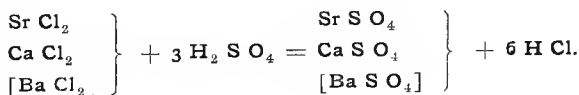
(b) The same flame may be examined by the spectroscope. (See paragraph 94.)

(c) Dissolve the remainder of the precipitate in water, and filter. To the *perfectly clear* filtrate, add a few drops of *pure* Chlorohydric acid, and then, aqueous solution of Calcic sulphate, ( $\text{CaSO}_4$ ); a white precipitate of Baric sulphate indicates presence of Barium.

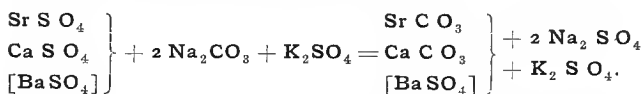


### *The Dissolved Portion, Tested for Strontium and Calcium.*

98. To the alcoholic filtrate, add dilute Sulphuric acid. This produces a white, gelatinous precipitate of Strontic and Calcic sulphates (together with a small amount of Baric sulphate—afforded by the traces of Baric chloride, which the alcohol inevitably takes up). Filter the mixture.

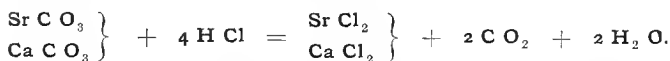


99. Transfer the precipitate to a casserole, and boil it for five minutes in a mixture of solutions of Sodid carbonate and Potassic sulphate. By this operation, Strontic sulphate is changed to Strontic carbonate; Calcic sulphate is changed to Calcic carbonate; but the Baric sulphate remains unchanged (the Potassic sulphate being the agent which is employed for the purpose of preventing the change, in the case of Baric sulphate).



Filter; and *thoroughly wash the precipitate with hot water*. This is for the purpose of removing all Sodid and Potassic sulphates; it is a step of vital importance to the success of the later processes.

100. Transfer the precipitate from the filter to a *test-tube*. Add a few drops of *pure* Chlorohydric acid. The precipitate *should* dissolve with effervescence.



Filter again. Evaporate the solution almost to dryness. Dissolve the residue in water. Filter yet again.

(We have now completely separated and removed all Barium; and we have the Strontium and Calcium as chlorides.)

101. Divide the solution into two parts.

**First Portion.**—To this, add solution of Calcic sulphate. A white precipitate, appearing either immediately or after a few minutes' application of heat, indicates presence of Strontium.



(Of course the *first portion* should not be tested for Calcium, since we have *added* Calcium sulphate to it.)

**Second Portion.**—If *Strontium* has been found in the first portion, add Potassic sulphate. This produces both Strontium sulphate and Calcium sulphate.



Now, although this process precipitates all the Strontium, and part of the Calcium, yet a small quantity of Calcic sulphate (sufficient for its detection) remains in solution.

**102.** Filter, and to the *perfectly clear* filtrate, add Ammonic hydrate and Ammonic oxalate,  $(\text{NH}_4)_2\text{C}_2\text{O}_4$ . A white precipitate of Calcic oxalate appears, if Calcium is present.



NOTE. If, by the test of the first portion, Strontium was shown to be *absent*, then the second portion may be at once tested for Calcium—and this without addition of Potassic sulphate—the main purpose of the latter reagent, here, being to throw Strontium, when present, out of the solution.

### *Additional Experiment.*

**103.** To a water solution of Calcic sulphate ( $\text{CaSO}_4$ ), add Ethylic alcohol; the Calcic sulphate appears as a white precipitate, because the mixture of alcohol and water, cannot hold dissolved as much of the salt, as water alone can.

**104. Common Forms of Fourth Group Metals.**

Muriate of Baryta,	Baric chloride; white,	$\text{Ba Cl}_2 + 2 \text{H}_2 \text{O}.$
Nitrate of Baryta,	Baric nitrate; white,	$\text{Ba (N O}_3)_2.$
Heavy spar, natural	Baric sulphate; white,	$\text{Ba S O}_4.$
Muriate of Strontia,	Strontic chloride; white,	$\text{Sr Cl}_2 + 3 \text{H}_2 \text{O}.$
Nitrate of Strontia,	Strontic nitrate; white,	$\text{Sr (N O}_3)_2.$
Quick-lime,	Calcic oxide; grayish white,	$\text{Ca O}.$
Slaked lime,	Calcic hydrate; white,	$\text{Ca O}_2 \text{H}_2.$
Chalk, marble, lime- stone, etc.,	Calcic carbonate,	$\text{Ca C O}_3.$
Gypsum, plaster-of- Paris, land-plaster,	Calcic sulphate; white,	$\text{Ca S O}_4.$
Bone-ash, mostly	Calcic phosphate,	$\text{Ca}_3 (\text{P O}_4)_2.$
Bleaching powder, a mixture of	Calcic hypo-chlorite, Calcic chloride and Calcic hydrate,	$\text{Ca O}_2 \text{Cl}_2 + \text{Ca Cl}_2 + \text{Ca O}_2 \text{H}_2.$



CHAPTER V.  
FIFTH GROUP OF METALS.

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*Description.*

**105.** The Fifth Group includes those metals which are *left in the solution*, after the precipitation and removal of the members of the preceding Groups.

The metals are—

Magnesium,	Mg,		II.
Potassium,	K,	(Kalium),	I.
Sodium,	Na,	(Natrium),	I.
Ammonium,	N H <sub>4</sub> ,		I.

(Ammonium is a compound radicle; it is called the hypothetical metal, because it acts chemically the part of a metal, in forming salts, etc.)

*Types of Compounds.*

Magnesic,	Mg Cl <sub>2</sub> .
Potassic,	K Cl.
Sodic,	Na Cl.
Ammonic,	N H <sub>4</sub> Cl.

*Remarks.*

NOTE I. The reason for the above-mentioned metals belonging to this Group, is evidently to be found in the fact that they decline to form insoluble compounds. This peculiarity prevents our detecting them by such a systematic and orderly process as is applicable to the other Groups.

NOTE II. It is obvious that our tests for Ammonium, should *not* be applied to the filtrate from the preceding Groups. For this filtrate is certain to contain salts of Ammonium (from compounds used in the detection of the metals of the Third and Fourth Groups), whether Ammonium salts were present at the beginning or not.

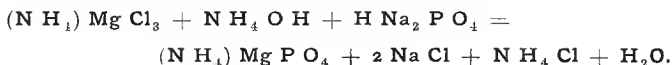
The tests for Ammonium must be applied to a portion of the *original solution*, reserved for the purpose.

### *The Process.*

106. The filtrate from the Fourth and preceding Groups must be divided into *three parts*, and treated as follows :

#### *The Test for Magnesium.*

107. **First Portion.**—To this portion add first Ammonic hydrate to alkaline reaction; then add Hydro-disodic phosphate ( $\text{HNa}_2\text{PO}_4$ ). A white precipitate of Ammonio-magnestic phosphate ( $\text{NH}_4\text{MgPO}_4$ ) constitutes the test. The precipitate, especially if relatively small in quantity, assumes a distinct crystalline form upon being left to itself for a day or two.



#### *The Test for Sodium (and Potassium).*

108. **Second Portion.**—Evaporate this portion, almost to dryness. Dip a cleaned platinum wire into the residual drop of liquid, and try it in a lamp-flame with the spectroscope.

109. Sodium is always detected. (It is always distributed throughout the atmosphere as some salt of Sodium, probably  $\text{NaCl}$ .) When the spectroscope reveals only a pale orange line, the Sodium must be considered as merely atmospheric. When at the line D, there appears

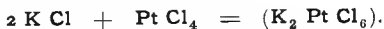
a dazzling orange band, it must be considered the test for Sodium, and as showing the *presence of Sodium in the solution under examination.*

**110.** The appearance of a rich, dark-red line at the left of the Sodium line (near line A), and also, of a diffused spectrum-light through the whole field, and also, of a violet line (at the other end of the spectrum, near H, and difficult of recognition), indicates *presence of Potassium.* (If much Sodium is present, the Potassium spectrum is difficult of recognition with ordinary laboratory spectroscopes.)

**111.** When but one of these metals is present, the color imparted to the flame is very characteristic, so that the metal may be detected without using the spectroscope. *Sodium* gives a strong orange color. *Potassium* alone, gives a delicate pinkish-violet shade. For the establishing of this latter shade by comparison, dip another clean platinum wire in pulverized Potassic nitrate; then hold it in the flame. The proper Potassium flame readily appears.

### *The Test for Potassium.*

**112. Third Portion.**—Evaporate this portion to dryness. Then carefully heat the residue to redness. This is to expel Ammonium salts. Cool the vessel. Dissolve the residue in two or three drops of water, and filter through a very small filter. To the filtrate, add a few drops of Platinic chloride ( $\text{PtCl}_4$ ). A yellow, granular, and crystalline precipitate of Potassio-platinic chloride constitutes the test for Potassium.



(A similar precipitate of  $(\text{N H}_4)_2 \text{ Pt Cl}_6$  is afforded by Ammonium salts. Hence, in order to be certain that a given precipitate is an indica-

cation of Potassium, we must have previously made sure of the removal of all Ammonium compounds.)

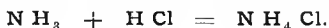
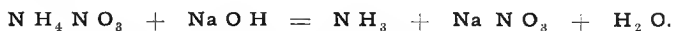
### *The Test for Ammonium.*

**113. Portion of Original.**—To a portion of the original solution, add Sodid hydrate (NaOH). If Ammonium is present, Ammonia gas (NH<sub>3</sub>) will be liberated. This gas may be recognized—

(a) By its pungent odor;

(b) By its giving a blue color to *moist*, red litmus-paper *suspended in the gas* (but not touching the walls of the test-tube);

(c) By giving heavy, white fumes of NH<sub>4</sub>Cl when a rod dipped in Chlorohydric acid is suspended in the tube containing the escaping gas. (In this latter case, judgment must be exercised, since *concentrated* Chlorohydric acid gives of itself a *fume* upon exposure to moist air.)

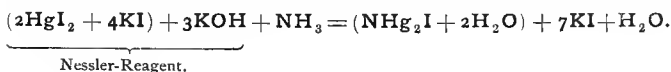


### *Nessler's Test for Ammonium.*

**114.** In a clean retort or side-neck test-tube, place a very small amount of water and a short stick of solid Caustic Soda (NaOH). Connect the apparatus with a test-tube used as a receiver, the latter containing a small amount of water and some Nessler-reagent (described below). Now bring the contents of the retort to full ebullition, for the purpose of making sure that the apparatus and reagents are free from Ammonia. This being assured, add to the



contents of the retort a small portion of the substance to be tested (either in the solid or in the liquid form). Then, again bring the contents of the retort to active boiling. If Ammonia is present, it should distill into the Nessler-reagent, and produce a brown precipitate of Di-mecur-ammonium iodide, thus :



### *Remarks on Nessler's Test.*

(a) Prepare Nessler-reagent as follows. Dissolve  $17\frac{1}{2}$  grammes of Potassic iodide in a small amount of distilled water. Add, little by little, to this, a strong aqueous solution of Mercuric chloride (Corrosive sublimate,  $\text{Hg Cl}_2$ ), until the precipitate, at first formed, fails to re-dissolve. Filter, and to the filtrate add a solution of 80 grammes of stick potash (Potassic hydrate,  $\text{K O H}$ ). Dilute the whole to 500 cubic centimetres. Finally, add about  $2\frac{1}{2}$  cubic centimetres of a concentrated solution of Mercuric chloride. Let the solution stand, to subside. Decant the clear, for use. (*Wanklyn.*)

(b) In certain cases, the Nessler test may be applied at once to the original solution; but it is always preferable to distill as above described, since by this operation the Ammonia is separated from other substances (for example, Sulphides) which would interfere with the action of the test.

(c) It should not be forgotten that there are substances, containing Nitrogen and Hydrogen, not already combined in the form of Ammonium. Some of these yield Ammonia gas by the treatment described in the text.

### *Additional Experiments.*

**115.** Add a few drops of solution of Magnesian sulphate (Epsom salts,  $\text{MgSO}_4 + 7\text{H}_2\text{O}$ ) to about a pint of water. Next add Ammonic chloride, and then Ammonic hydrate, and finally, Hydro-di-sodic phosphate. Allow the whole to stand a few days. Distinct crystals of Ammonio-magnesian phosphate ( $\text{NH}_4\text{MgPO}_4 + 12\text{H}_2\text{O}$ ) will appear.

**116.** Burn a fragment of Magnesium wire.

**117.** With care, throw a fragment of Sodium (not bigger than a pin-head) on a wet filter-paper. The metal should burst into flame.

**118.** With great care, throw a fragment of Potassium (not bigger than a pin-head) on water in a deep beaker. The metal should burst into flame.

### 119. *Common Forms of Fifth Group Metals.*

Magnesia,	Magnesian hydrate and carbonate,	$\text{Mg O}_2 \text{ H}_2 + \text{Mg C O}_3.$
Epsom salts,	Magnesian sulphate,	$\text{Mg S O}_4 + 7 \text{ H}_2 \text{ O}.$
Potash, or Caustic potash,	Potassic hydrate,	$\text{K O H}.$
Pearlash, or Sal-tartar,	Potassic carbonate,	$\text{K}_2 \text{ C O}_3.$
Salt-petre, or nitre,	Potassic nitrate,	$\text{K N O}_3.$
Cream of Tartar,	Hydro-potassic tartrate,	$(\text{H K H}_2)\text{O}_4 (\text{C}_4 \text{ H}_2 \text{ O}_2).$
Common salt,	Sodic chloride,	$\text{Na Cl}.$
Caustic soda, or Soda by lime,	Sodic hydrate,	$\text{Na O H}.$
Soda ash, a commercial crude form of	Sodic carbonate,	$\text{Na}_2 \text{ C O}_3.$
Soda, or Sal-soda, or Soda crystals,	Sodic carbonate,	$\text{Na}_2 \text{ C O}_3 + 10 \text{ H}_2 \text{ O}.$
Glauber's salt,	Sodic sulphate,	$\text{Na}_2 \text{ S O}_4 + 10 \text{ H}_2 \text{ O}.$
Chili salt-petre,	Sodic nitrate,	$\text{Na N O}_3.$
Ammonia,	Ammoniac hydrate solutions,	$\text{N H}_4 \text{ O H}.$
Sal-ammoniac,	Ammoniac chloride,	$\text{N H}_4 \text{ Cl}.$

## CHAPTER VI.

### TREATMENT OF UNCOMBINED METALS AND METALLIC ALLOYS.

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**120.** THE uncombined metals are divided into three classes, the classification depending upon their relations to Nitric acid.

*Class First.*—Those metals which are unaffected by  $\text{HNO}_3$ : *Gold and Platinum (and the Platinum Metals)*.

*Class Second.*—Those metals which are oxidized by  $\text{HNO}_3$ , forming insoluble compounds: *Antimony and Tin*.

*Class Third.*—Those metals which are oxidized by  $\text{HNO}_3$ , forming soluble Nitrates. This includes all the metals, except those of the two preceding classes.

#### ***Process.***

**121.** To the metal or alloy, which should be as finely divided as possible, add Nitric acid of medium strength, and warm the whole if necessary. Decant the dissolved part upon a filter. Treat the insoluble part anew with Nitric acid, and decant again upon the same filter as before. Next, boil in water the part which still remains insoluble; then transfer solution and sediment to the filter already used. Set the *filtrate* aside for future use, as described in paragraphs 123, 124.

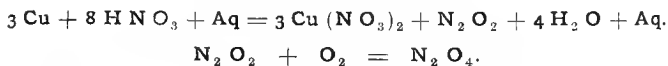
122. Wash the *precipitate* thoroughly with boiling water, to remove all soluble matters. Throw the washings away; but examine the precipitate, as described in paragraphs 125-127.

### *Detection of Metals that Dissolve in Nitric Acid.*

123. Now evaporate the filtrates (paragraph 121) to dryness; then re-dissolve the residue in water and a drop or two of Nitric acid.

124. The solution may contain any metals except *Gold* and *Platinum*, *Antimony* and *Tin* (though traces of the latter two are sometimes dissolved); it is tested for the metals according to the processes described under the various Groups. It may be observed that the only metals which can be said to be of common occurrence in alloys, are those of the *First* and *Second Groups*, and *Nickel* and *Zinc* of the *Third Group*, though, of course, other metals *may* be present.

#### *Example of Reactions.*

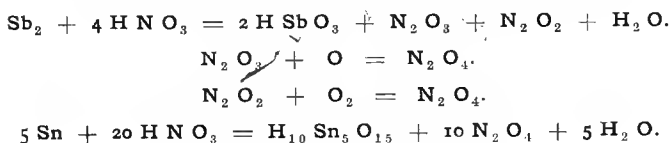


NOTE. In alloys containing a large amount of Gold or Platinum, Nitric acid fails to dissolve the base metal—even when such base metal is ordinarily dissolved with ease by  $\text{H N O}_3$ . This difficulty is observed when the alloy contains more than *one part* of noble metal, to *three parts* of base metal. When this difficulty is suspected, a portion of the alloy may be fused with so much Silver as will make three-fourths of the new alloy thus formed. The new alloy may then be treated as above described. If we desire to test for Silver itself, this may be accomplished by fusing a new portion of the original alloy with Zinc—or some other suitable metal—and then parting it, as before, with Nitric acid.

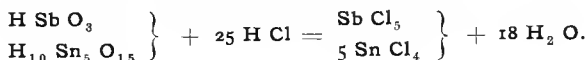
### *The Residue, Insoluble in Nitric Acid.*

**125.** If the precipitate is dark-colored, it probably contains Gold or Platinum, or both. In any case, boil it for some time in concentrated Chlorohydric acid; then dilute considerably with water, and boil again.

#### *Action of Nitric Acid, on Antimony and Tin.*



#### *Action of Chlorohydric Acid.*

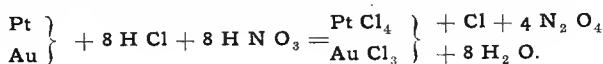


### *The Tests for Antimony and Tin.*

**126.** Filter, and test the filtrate (paragraph 125) for Antimony and Tin, by the Zinc-and-Platinum process, as described on pages 20–22. Test the undissolved residue for Gold and Platinum.

### *The Examination for Gold and Platinum.*

**127.** Dissolve this residue in *Aqua-regia*. Then evaporate the solution, nearly to dryness.



### *The Test for Gold.*

Dissolve some Stannous chloride ( $\text{SnCl}_2$ ) in water and Chlorohydric acid. To the solution, add enough Ferric

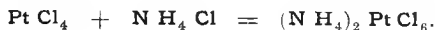
chloride ( $\text{Fe}_2\text{Cl}_6$ ) to afford a faint yellow color to the mixture.



To this liquid, add one drop of the *Aqua-regia* solution which has been prepared. A very fine precipitate, of a dark-brown or a purple color, and known as *Purple of Cassius*, indicates the presence of Gold.

### *The Test for Platinum.*

To the remainder of the *Aqua-regia* solution, add solution of Ammonic chloride ( $\text{NH}_4\text{Cl}$ ). If much Platinum is present, an immediate separation of a yellow precipitate of Ammonio-platinic chloride appears. It is always advisable to allow the mixture to evaporate spontaneously in a watch-glass; upon adding water to the residue, the Ammonio-platinic chloride remains crystalline and insoluble.



## CHAPTER VII.

### DETECTION OF ACIDS AND ACID RADICLES.

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**128.** ALTHOUGH a salt is usually spoken of as containing an acid, it contains only the representative of that acid. Thus, common salt (NaCl) certainly contains no Chlorohydric acid (HCl); but the Chlorine in the salt is the representative of the acid mentioned. So, Potassic nitrate ( $\text{KNO}_3$ ) certainly contains no Nitric acid ( $\text{HNO}_3$ ); but it contains  $\text{NO}_3$ , which is a group of elements contained in Nitric acid, and which group is the representative of Nitric acid.

But it has arisen—from the inconvenience attending the use of such terms as *representative*, *acid radicle*, etc.—that chemists commonly speak of salts as containing acids. Thus, sulphates are spoken of as containing Sulphuric acid, phosphates as containing Phosphoric acid, and so on.

**129.** The more common acid radicles are divided into six Groups.

In testing for acid radicles, no strictly continuous processes of isolation from a single portion of substance can be used; most of the tests are performed upon separate portions of the original substance, set aside for this use.

## FIRST GROUP OF ACIDS.

## THE DEFLAGRATING ACIDS.

**130.** This group includes those acids whose salts readily deflagrate, when heated on charcoal.

It consists of—

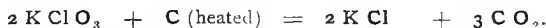
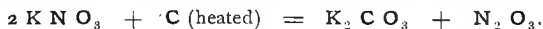
Nitric acid,  $\text{H N O}_3$ .

Chloric acid,  $\text{H Cl O}_3$ .

*Preliminary Tests.*

**131.** Heat upon charcoal a portion of the dry salt. (If the substance is not a salt, evaporate a portion of the solution to dryness, and test the residue.) If deflagration ensues, it indicates the probable presence of a Nitrate or a Chlorate.

The deflagration is *rapid combustion* of the coal, due to the fact that the salts, in question, contain large amounts of oxygen, held by feeble affinity.



**132.** Having carefully made the general group test, proceed to test for each of the acids as follows.

*NITRIC ACID (H N O<sub>3</sub>), IN NITRATES.*

NOTE. All of the salts of Nitric acid are soluble in water.

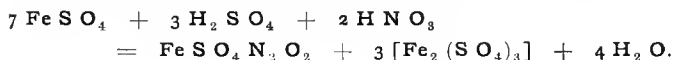
*Test A.—The Copperas Test.*

**133.** To the solution, supposed to contain a Nitrate, add pure; concentrated Sulphuric acid, to liberate the Nitric acid. (If a precipitate appears, its presence may obscure the test; allow it, therefore, to subside, and then decant



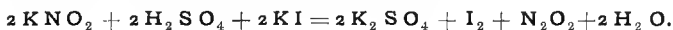
the clear liquid; test the liquid as follows.) Add one or two crystals of Ferrous sulphate ( $\text{FeSO}_4$ ). If Nitric acid is present, the solution around the crystals becomes brown, from the formation of Ferrous nitro-sulphate ( $\text{FeSO}_4\text{N}_2\text{O}_2$ ).

**134.** Ferrous sulphate has a strong affinity for oxygen; one portion can withdraw from free Nitric acid a part of its oxygen, leaving  $\text{N}_2\text{O}_2$ , with which another portion of Ferrous sulphate can unite to form  $\text{FeSO}_4\text{N}_2\text{O}_2$ .



**Test B.—The Iodo-Starch Test.\***

**135.** Make the solution to be tested, slightly acid with dilute  $\text{H}_2\text{SO}_4$ ; test it with litmus-paper. Add a fragment of metallic Zinc. Boil the whole, for about five minutes, in a covered beaker. A portion of the Nitrate, if any is present, is thus reduced to a Nitrite (thus, from  $\text{KNO}_3$  or  $\text{HNO}_3$  to  $\text{KNO}_2$  or  $\text{HNO}_2$ ). Filter. To the filtrate add a few drops only, of a cold solution containing Potassic iodide and starch. The blue or purple coloration of the Iodide of starch constitutes the test.



**FREE OR UNCOMBINED NITRIC ACID ( $\text{HNO}_3$ ).**

**136.** Test the original solution, by adding it to a faintly-blue solution of Indigo sulphate. Free Nitric acid bleaches the latter. (So also does free Chlorine, and some of its compounds; and also Iodine, Bromine, and a few other substances.)

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\* *F. H. Storer*, CHEMICAL NEWS, 1877, vol. 36, pp. 115 and 133; especially, *footnote* to p. 117.

**137.** In another portion of the original solution, boil some white fragments of silk or wool or quill; free Nitric acid turns them yellow. (So also does Picric acid.)

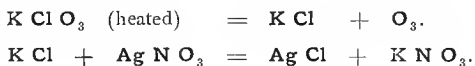
**138.** Free Nitric acid, also, when in considerable quantity, affords the Copperas test, with crystal of  $\text{FeSO}_4$  before any Sulphuric acid is added.

### CHLORIC ACID ( $\text{H Cl O}_3$ ), IN CHLORATES.

NOTE. Owing to its instability, Chloric acid ( $\text{H Cl O}_3$ ) rarely occurs uncombined. Only one compound of it is at all common,  $\text{K Cl O}_3$ .

**139.** In absence of Nitrates, deflagration on charcoal shows probable presence of a Chlorate.

The decisive test depends upon the fact that, at a low red heat, Chlorates change into *Chlorides*, which Chlorides produce an abundant precipitate with Argentic nitrate, while the original Chlorates give no precipitate, or almost none.



**140.** To detect a Chlorate, therefore, proceed as follows. Heat the substance on porcelain, and, after cooling, dissolve the residue in water. Now test for Chlorides by adding Argentic nitrate and Nitric acid. If a precipitate of Argentic chloride ( $\text{AgCl}$ ) appears, it shows the presence of a Chloride.

But we have yet to decide whether this Chloride was really the result of the decomposition of a Chlorate, or whether it was *originally* present. For this purpose, test the original solution for Chlorides, by means of Argentic nitrate and Nitric acid.

(a) If a Chloride is *not* thus detected in the original substance, it proves that the Chloride discovered previously, was actually the result of the decomposition of a *Chlorate* originally present.

(b) But if a Chloride *is* thus detected in the original substance, it is necessary to add to a portion of the original solution, in presence of Nitric acid, just enough Argentic nitrate to precipitate *all* the Chlorine which is present as a Chloride. Filter, and neutralize the filtrate with pure Sodic hydrate (NaOH), to retain the Chloric acid; evaporate to dryness, and ignite, and then test the residue for Chlorides. Any Chloride *now* detected must have arisen from the decomposition of a *Chlorate* in the original solution (since by the preceding operations all other Chlorine has been removed).

#### 141. *Common Forms of First Group Acids.*

Aqua fortis, or parting acid,	Nitric acid,	$\text{H N O}_3$ .
Nitre, or saltpetre,	Potassic nitrate,	$\text{K N O}_3$ .
Soda saltpetre, or Chili saltpetre,	Sodic nitrate,	$\text{Na N O}_3$ .
Chlorate of potash,	Potassic chlorate,	$\text{K Cl O}_3$ .



## CHAPTER VIII.

### SECOND GROUP OF ACIDS.

#### *THE EFFERVESCING ACIDS.*

**142.** THIS Group includes those acids whose salts liberate gases upon the addition of stronger acids, such as dilute Sulphuric acid, dilute Chlorohydric acid, and, in some cases, dilute Nitric acid. The gases liberated, are either the acid in question or its anhydride.

For the purpose of producing the most distinct effervescence, the test is best applied to the dry salt—or at least to the dry salt pulverized, and slightly moistened with water.

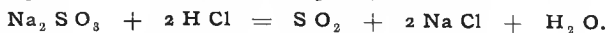
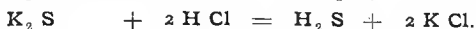
This Group includes—

Carbonic acid,	$\text{H}_2 \text{C O}_3$
Sulphyhydric acid,	$\text{H}_2 \text{S}$ .
Sulphurous acid,	$\text{H}_2 \text{S O}_3$ .
Cyanohydric acid,	$\text{H C N}$ , ( $\text{H Cy}$ ).

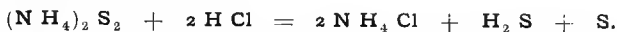
#### *The Preliminary Test, with Chlorohydric Acid.*

**143.** To a portion of the solid substance, slightly moistened with water, add a few drops of dilute *Chlorohydric acid*. A variety of effects may be observed.

(a) Effervescence indicates presence of one of the before-mentioned acids.



(b) A white, milky precipitate of Sulphur, is often produced from sulphides.



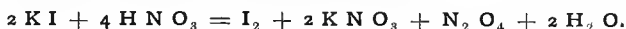
(c) A gelatinous and very thick precipitate, of Silicic acid, is often produced from soluble silicates.



### *The Test with Nitric Acid.*

144. The foregoing experiment may be repeated, only using *Nitric acid* in place of Chlorohydric acid. A variety of effects, such as those just mentioned, (a), (b), and (c), may be observed. In addition, the following may be observed:

(d) A brown or black precipitate, or coloration, may be an indication of the presence of Iodine.



(e) A white precipitate may be due to the separation of Stannic acid from Stannates, thus:



(or to the separation of Antimonic acid from Antimonites, thus:



145. Having applied these preliminary tests, proceed to make a special test for each of the acids as follows.

### **CARBONIC ACID ( $\text{H}_2 \text{C O}_3$ ), IN CARBONATES.**

(Observe that at the ordinary atmospheric pressure and temperature, Carbonic acid ( $\text{H}_2\text{CO}_3$ ) decomposes into Carbonic anhydride ( $\text{CO}_2$ ) and water ( $\text{H}_2\text{O}$ ).

In a small test-tube, place a portion of the original material (preferably in the form of the finely-pulverized solid). To it, add a few drops of Nitric acid. The Carbonic gas should appear with effervescence and fill the test-tube with invisible gas. Slowly, pour this gas—although invisible—into another test-tube, which is two-thirds filled with lime-water. A white, cloudy precipitate of Calcic carbonate ( $\text{CaCO}_3$ ) should appear on the surface of the lime-water; thus:



NOTE I. This test is liable to fail from use of old Lime-water. The solution tends to lose its Lime by gradual absorption of Carbonic gas from the atmosphere, and consequent precipitation of all of the Calcium as Calcic carbonate.

In case of doubt, take a portion of the Lime-water on hand, and test it with known Carbonic gas from a known carbonate. If it will not serve for the test in this case, of course it is not suitable for use in testing the problematical substance under examination.

NOTE II. The test is made to better advantage by generating the gas in a side-neck test-tube or side-neck flask, and conducting the gas by a rubber-tube into the Lime-water.

### **SULPHYDRIC ACID ( $\text{H}_2 \text{ S}$ ), IN SULPHIDES.**

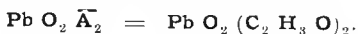
**146.** To a portion of the original solid or solution, add dilute Sulphuric acid. This should liberate  $\text{H}_2 \text{ S}$  from many of its salts, thus:



If much  $\text{H}_2 \text{ S}$  is set free, it may be recognized by its odor.

As an additional test, cover the tube from which the gas is escaping, with a piece of *lead-paper* (prepared by moistening filter-paper with Plumbic acetate, and adding to the wet part, a drop of Sodid hydrate). If the gas is

$H_2S$ , it will betray itself by forming black Plumbic sulphide ( $PbS$ ) on the lead-paper.



NOTE. In case of some sulphides, especially those of metals of the First and Second Groups, the test as above described is inoperative, owing to the fact that the sulphides in question are not readily decomposed by dilute Sulphuric acid. The same difficulty arises in case of many compact mineral sulphides.

In these cases the sulphur is detected by other means, as, for example, it makes its appearance as free sulphur, when the sulphide is treated with *Aqua-regia*, or with *Nitric acid*, or when the substance is heated in a tube closed at one end. Sometimes it is detected as an alkaline sulphide when the original substance is fused with an alkaline carbonate.

## **SULPHUROUS ACID ( $H_2 S O_3$ ), IN SULPHITES.**

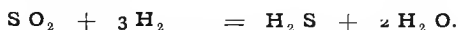
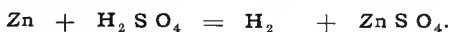
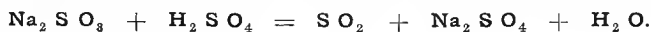
### *First Test.*

147. To a portion of the original substance, add dilute Sulphuric acid. If a gas escapes, observe the odor. Sulphites yield Sulphurous anhydride ( $SO_2$ ), a gas which has the choking odor of burning sulphur—often observed when a sulphur match commences to burn. If the odor is not immediately apparent, the gas may often be expelled in greater quantity by boiling the mixture.

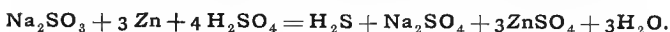
### *Second Test.*

148. To another portion of the original substance, add a fragment of metallic Zinc and a few drops of dilute Sulphuric acid. If the solution contains a sulphite, Sulphurous anhydride ( $SO_2$ ) will be liberated—but the hydrogen, which is also escaping, will reduce  $SO_2$ , forming  $H_2O$  and

H<sub>2</sub>S. The latter may be recognized by its odor, or by the lead-paper as described under the heading Sulphydric acid, page 64.



The above reactions, combined, give the following:



### *Third Test.*

**149.** To a portion of the original substance, add some pure Nitric acid; this will, indeed, decompose a part of the Sulphite, liberating SO<sub>2</sub>, but it will also oxidize a part, forming a Sulphate (*e. g.*, changing K<sub>2</sub>SO<sub>3</sub> into K<sub>2</sub>SO<sub>4</sub>). Now add Baric chloride (BaCl<sub>2</sub>), as in testing for Sulphates. A white precipitate, of Baric sulphate, constitutes the test.



NOTE. It will be observed that the Second and Third tests are indirect tests, dependent upon the change of sulphites into sulphides or into sulphates.

Before relying on either of these tests, we must make certain, *first*, that there is not present any sulphur compound—capable of these changes—other than a sulphite; *secondly*, that sulphides or sulphates are not themselves present when the test commences.

### *CYANOHYDRIC ACID (HCy), IN CYANIDES.*

**150.** This acid, although often made evident by the preliminary tests for this Group of effervescing acids, is properly detected by the processes for the Silver Group (which see, page 68).



**151. Common Forms of Second Group Acids.**

Plain soda water,		$\text{H}_2 \text{C O}_3$ with water.
Marble,	} Calcic carbonate,	$\text{Ca C O}_3$ ,
Limestone,		
Chalk,		
	and other carbonates.	
Sulphuretted hydrogen gas,		$\text{H}_2 \text{S}$ , as a gas or dissolved in water.
Sulphurous acid,		$\text{H}_2 \text{S O}_3$ with water.
Antichlor,	Sodic sulphite,	$\text{Na}_2 \text{S O}_3$ .



## CHAPTER IX.

### THIRD GROUP OF ACIDS.

#### *THE SILVER GROUP.*

**152.** THIS Group includes those acids which produce precipitates upon addition of Argentic nitrate ( $\text{AgNO}_3$ ) in presence of Nitric acid.

The acids are—

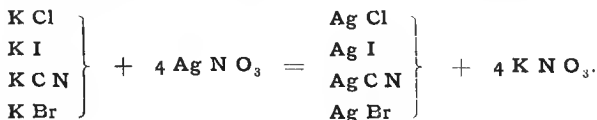
Chlorohydric acid,	H Cl.
Cyanohydric acid,	H Cy (H C N).
Iodohydric acid,	H I.
Bromohydric acid,	H Br.

#### *The Process.*

**153.** To the solution, supposed to contain any of the above acids or their representatives, add Argentic nitrate and Nitric acid. If a precipitate is formed, shake it thoroughly to make it collect.

The precipitate may contain one or more of the following compounds;

Argentic chloride,	Ag Cl,	white.
Argentic cyanide,	Ag Cy (Ag C N),	yellowish white.
Argentic iodide,	Ag I,	yellowish white.
Argentic bromide,	Ag Br,	yellowish white.



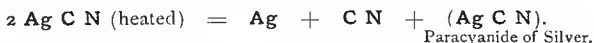
**154.** Filter; then remove the precipitate, and digest it in Ammonic hydrate. This reagent dissolves the Argentic chloride and the Argentic cyanide, but it leaves the Argentic iodide and the Argentic bromide undissolved.

In case, therefore, of a precipitate, undissolved by Ammonic hydrate, test for iodides and bromides, as described in paragraphs 157 and 158.

Filter, and test the filtrate as follows:

### **CHLOROHYDRIC ACID (HCl), IN CHLORIDES.**

To this filtrate, add Nitric acid, to acid reaction. This reagent (HNO<sub>3</sub>) neutralizes the solvent power of the Ammonic hydrate; if now a precipitate appears, it consists of Argentic chloride or cyanide, or both. Remove the precipitate to a piece of porcelain, and ignite until the precipitate fuses. The fusion decomposes the AgCy (=AgCN), but does not injure the Argentic chloride.



**155.** To the fused mass, supposed to consist mainly of Argentic chloride (AgCl) and metallic Silver, add a bit of Zinc and some dilute H<sub>2</sub>SO<sub>4</sub>; the hydrogen, thus evolved, decomposes the fused AgCl, forming HCl and leaving metallic Silver.

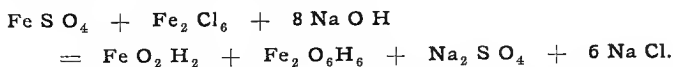


Filter, and to the filtrate add Argentic nitrate and Nitric acid. The appearance of a white precipitate of Argentic chloride indicates the presence of Chlorohydric acid, or some other Chloride, in the original solution.

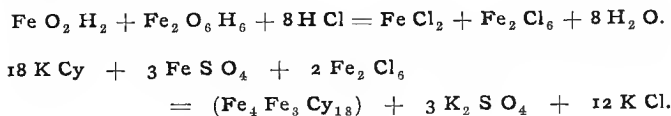
### **CYANOHYDRIC ACID (HCy), IN CYANIDES.**

**156.** Now return to a portion of the original solution, and test whether some cyanide was not also present.

For this purpose, add to the solution Sodid hydrate (to alkaline reaction), and then a mixture of Ferrous and Ferric salts (*e. g.*,  $\text{FeSO}_4$  and  $\text{Fe}_2\text{Cl}_6$ ). At first, there appears a dingy precipitate of Ferrous hydrate ( $\text{FeO}_2\text{H}_2$ ) and Ferric hydrate ( $\text{Fe}_2\text{O}_6\text{H}_6$ ).



Upon adding a little Chlorohydric acid ( $\text{HCl}$ ), the hydrates dissolve, and there appears the blue precipitate of Ferric ferro-cyanide ( $\text{Fe}_4\text{Fe}_3\text{Cy}_{18}$ ), called Prussian blue, which shows the presence of a cyanide in the original solution.



### ***IODOHYDRIC ACID (HI), IN IODIDES.***

**157.** Now return to the original solution, and test for Iodine as follows. To the somewhat concentrated solution, add a few drops of Nitric acid; this reagent liberates Iodine. Now gradually add this solution of Iodine to a tube containing some very thin starch-paste. The formation of a beautiful blue precipitate of Iodide of starch constitutes the test for an Iodide.

NOTE I. Make the starch-paste *very thin*, by boiling a minute fragment of starch in a considerable quantity of water. Cool this liquid.

NOTE II. The blue Iodide of starch disappears upon heating a liquid containing it. It re-appears upon cooling.

NOTE III. Observe that presence of Ethylic alcohol interferes with the production of Iodide of starch, and it may be entirely preventive when but little Iodine is present.

**BROMOHYDRIC ACID (H Br), IN BROMIDES.**

**158.** To the original solution, add a few drops of Chlorine-water. This should liberate Bromine, and should impart a yellow color to the solution.



Now add to the solution, a few drops of Chloroform or of Carbon di-sulphide. These solvents should gather the Bromine from the water, and acquiring a deeper color, should carry it with themselves to the bottom of the vessel.

NOTE I. The test just given is not applicable in presence of Iodine or Iodides, since these produce *nearly* the same effects as Bromine and Bromides. The detection of Bromine in presence of Iodine is somewhat difficult.

NOTE II. The Chlorine-water may be prepared by passing a little Chlorine-gas into water.

A convenient substitute for Chlorine-water is prepared as follows. To a single crystal of Potassic chlorate add two drops of Chlorohydric acid; now, *carefully* heat the mixture, and as soon as effervescence commences dilute the whole largely with water.

**159. Common Forms of Third Group Acids.**

Muriatic acid,	Chlorohydric acid,	H Cl.
Common salt,	Sodic chloride,	Na Cl.
Many chlorides.		
Prussic acid,	Cyanohydric acid,	H Cy (H C N).
Cyanide of potassium,	Potassic cyanide,	K Cy.
Iodide of potassium,	Potassic iodide,	K I.
Bromide of potassium,	Potassic bromide,	K Br.



## CHAPTER X.

### FOURTH GROUP OF ACIDS.

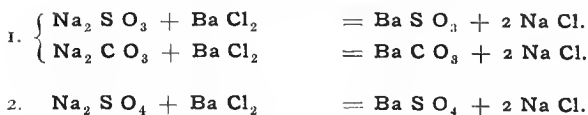
#### *THE BARIUM GROUP.*

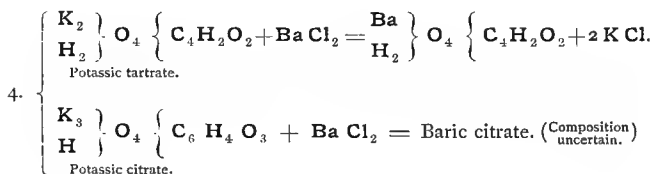
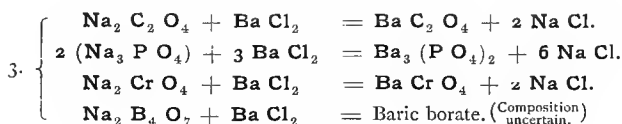
**160.** THIS Group includes those acids—either when free or in combination—which readily form insoluble salts, upon addition of Baric chloride to their water solution.

The acids are—

1.	$\left\{ \begin{array}{l} \text{Sulphurous acid,} \\ \text{Carbonic} \quad \text{“} \end{array} \right.$	$\left. \begin{array}{l} \text{H}_2 \text{ S O}_3 \text{ or S O}_2. \\ \text{H}_2 \text{ C O}_3 \text{ or C O}_2. \end{array} \right.$
2.	Sulphuric “	$\text{H}_2 \text{ S O}_4.$
3.	$\left\{ \begin{array}{l} \text{Oxalic} \quad \text{“} \\ \text{Phosphoric} \quad \text{“} \\ \text{Chromic,} \quad \text{“} \\ \text{Boracic} \quad \text{“} \end{array} \right.$	$\left. \begin{array}{l} \text{H}_2 \text{ C}_2 \text{ O}_4. \\ \text{H}_3 \text{ P O}_4. \\ \text{H}_2 \text{ Cr O}_4. \\ \text{H}_3 \text{ B O}_3. \end{array} \right.$
4.	$\left\{ \begin{array}{l} \text{Tartaric} \quad \text{“} \\ \text{Citric} \quad \text{“} \end{array} \right.$	$\left. \begin{array}{l} \text{H}_2 \left( \text{C}_4 \text{ H}_2 \text{ O}_2 \right) \text{ O}_4. \\ \text{H}_3 \left( \text{C}_6 \text{ H}_4 \text{ O}_3 \right) \text{ O}_4. \end{array} \right.$

#### *Examples of Reactions.*



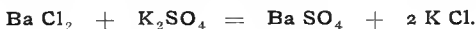


### SULPHITES AND CARBONATES.

161. With Baric chloride, these salts form respectively Baric sulphite ( $\text{BaSO}_3$ ) and Baric carbonate ( $\text{BaCO}_3$ ). But the latter salts are soluble in *dilute* and *pure* Chlorohydric acid. (See pp. 62-63-65.)

### SULPHURIC ACID ( $\text{H}_2 \text{S O}_4$ ), OR SULPHATES.

162. To the solution to be tested, add Baric chloride; if a fine, white precipitate, Baric sulphate ( $\text{BaSO}_4$ ), appears—and this precipitate does not re-dissolve in warm, *dilute*, *pure* Chlorohydric acid—it shows the presence of Sulphuric acid or a Sulphate.



### Free Sulphuric Acid.

163. Free Sulphuric acid may often be detected (in solutions known to contain its radicle) as follows. First, prove presence of a free acid by the reddening of litmus-paper. Then, place a portion of the original solution upon ordi-

nary writing-paper; finally, gently warm the paper, either at a safe distance above a lamp-flame, or in a water-bath, so that the moisture will dry off. If *free* Sulphuric acid is present, it will gradually concentrate upon the paper, so as to *blacken* it and rot it where the acid was placed.

Other free acids may rot the paper, but they do not *blacken* it, as free Sulphuric acid does.

NOTE.—Observe that the first test for Sulphuric acid cannot be made in presence of *concentrated* Chlorohydric acid. Thus, to some pure concentrated Chlorohydric acid, add solution of Baric chloride. An abundant white precipitate of Baric chloride (not Baric sulphate) appears. Decant the clear liquid, and to the white precipitate add pure water. The precipitate entirely dissolves, showing that it was not Baric sulphate.

### **OXALIC ACID ( $H_2 O_2 C_2 O_2$ ), OR OXALATES.**

#### ***Test A.—The Calcic Chloride Test.***

**164.** Before testing for Oxalic acid, any Sulphuric acid that is present must be removed as follows. Add sufficient pure Chlorohydric acid, to make the solution acid, and to hold in solution, salts other than Baric sulphate. Then add Baric chloride—in quantity just sufficient to precipitate all the Sulphuric acid radicle—a large excess being carefully avoided. Now filter the whole.

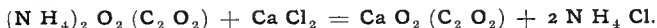
The filtrate may now contain Oxalic acid if it, or an Oxalate, was originally present. (But the filtrate will also contain some Chlorohydric acid and some Baric chloride—the amount of the latter depending on the care exercised in introducing it.)

**165.** To the filtrate, add Ammonic hydrate, to produce alkaline reaction. (There generally appears a precipitate of Baric oxalate containing all the Barium of the Baric



chloride added previously *in excess*; the precipitate also, of course, withdraws Oxalic acid—in amount proportional to the amount of Barium present; but the principal part of the Oxalic acid should remain in the solution.) Carefully filter to obtain a perfectly clear solution.

**166.** To the filtrate, add Calcic chloride ( $\text{CaCl}_2$ ); a white precipitate of Calcic oxalate,  $\text{CaO}_2(\text{C}_2\text{O}_2)$ , may appear. To the precipitate, after pouring off the clear liquid, add Acetic acid sufficient to produce acid reaction. The precipitate, if it is an Oxalate, should *not* dissolve.



NOTE I.—If, upon testing for Sulphuric acid, it is shown to be absent, it is better to take a new portion of the original material for testing for Oxalic acid. Ammonia and Calcic chloride can then be added at once.

NOTE II.—For further account of Oxalic acid, see pp. 85, 89, 91.

### *Test B.—The Ignition Test.*

**167.** In certain cases, this test may be applied at once to the original substance. It is always applicable to the precipitate of Calcic oxalate produced at the conclusion of *Test A*. (Paragraph 166.) It is based upon the fact that Oxalates (not Oxalic acid), by sufficient heating, change into Carbonates without signs of charring.

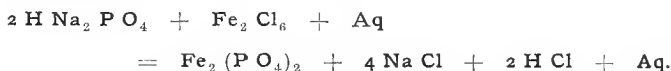


To apply this test, heat the dry substance on a fragment of porcelain, before the blowpipe. After cooling, test the residue carefully for a Carbonate; presence of the latter, under the conditions above-mentioned, indicates original presence of an Oxalate.

**PHOSPHORIC ACID ( $H_3PO_4$ ), OR PHOSPHATES.****Test A.—The Ferric Chloride Test.**

**168. First Case.**—In substances soluble in water, and not possessing acid reaction.

To the water solution, add Acetic acid, so as to give acid reaction. Then add a few drops of Ferric chloride ( $Fe_2Cl_6$ ). If a yellowish-white precipitate of Ferric phosphate  $Fe_2(PO_4)_2$  appears, it constitutes a test for Phosphoric acid.



NOTE. Presence of Acetic acid is necessary, to prevent the precipitation of *other* compounds of Iron; absence of stronger acids is necessary, since they would prevent the precipitation of the Ferric phosphate itself.

The presence of members of the First Group of Metals gives rise to precipitates of Chlorides of those metals, when Ferric chloride is added; but the *appearance* of these precipitates ought at least to indicate that they are other than Ferric phosphate.

**169. Second Case.**—In substances not soluble in water, but soluble in dilute acid; or in substances possessing acid reaction.

If the substance to be examined is insoluble in water, dissolve it in Chlorohydric acid. Next neutralize with Ammonic hydrate, as nearly as possible. If so much Ammonic hydrate has been added that the precipitate formed does not redissolve upon shaking, add Chlorohydric acid, drop by drop, until it does dissolve. Neutralize the excess of Chlorohydric acid by adding Ammonic acetate ( $NH_4O\bar{A}$ ) (prepared by adding, to Ammonic hydrate, Acetic acid to acid reaction). The Ammonic acetate neutralizes the free Chlorohydric acid; at the same time it sets free Acetic acid, which will hold in solution

any of the recently precipitated Phosphates, except Ferric phosphate.



To the clear solution, add Ferric chloride ( $\text{Fe}_2\text{Cl}_6$ ) and a little free Acetic acid; the characteristic precipitate of Ferric phosphate appears.

NOTE. If the precipitate of Ferric phosphate,  $\text{Fe}_2(\text{PO}_4)_2$ , appears upon addition of Ammonic acetate, it indicates the presence (in the original solution) of both Iron and Phosphoric acid; it then becomes unnecessary to add Ferric chloride.

### *Test B.—The Ammonic Molybdate Test.*

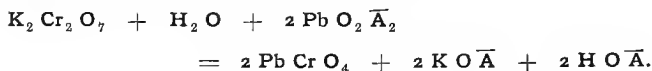
170. To a small quantity of the Nitric acid solution of Ammonic molybdate,  $(\text{NH}_4)_2\text{MoO}_4$ , add *a few drops* of the solution to be tested, pouring the latter down the side of the test-tube, so that it will float on the other solution.

If Phosphoric acid or a Phosphate is present, there appears a yellow precipitate of Ammonic phospho-molybdate. If the precipitate does not appear at once, add cautiously a little Ammonic hydrate; the yellow precipitate (which is soluble in excess of  $\text{NH}_4\text{OH}$ ) then appears at the plane of junction of the two solutions. A mere yellow coloration does not constitute a test.

### *CHROMIC ACID ( $\text{H}_2\text{CrO}_4$ ), OR CHROMATES.*

171. All the Chromates are colored, the neutral Chromates being generally yellow, while the Di-chromates are generally red. Hence, a colorless salt will not be likely to contain a Chromate.

To the solution to be tested, add Acetic acid and then Plumbic acetate; a yellow precipitate of Plumbic chromate ( $\text{PbCrO}_4$ , Chrome yellow) indicates presence of a Chromate.

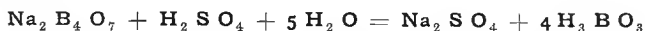


NOTE. Chrome yellow is soluble in concentrated Nitric acid ; also, in Sodic hydrate solution.

### **BORACIC ACID ( $\text{H}_3 \text{B O}_3$ ), OR BORATES.**

#### **Test A.—The Alcohol Test.**

**172.** This test for Borates is best applied to the dry salt, or to the residue from evaporation of the solution. To the salt, add concentrated Sulphuric acid—sufficient in quantity to decompose the salt—and then add Ethylic alcohol,  $(\text{C}_2\text{H}_5)\text{OH}$ . Now inflame the alcohol. The liberated Boracic acid will impart a green color to the flame. This color is best obtained by dipping a glass rod into the burning alcohol, and then withdrawing the rod. The flame, playing about the rod, affords an especially good test.



#### **Test B.—The Glycerine Test.\***

**173.** Finely pulverize the substance, and add one drop of Sulphuric acid to it. Moisten a platinum wire loop, with glycerine ; dip the wire into the powder. Now place the wire in the lamp-flame, for a moment. A distinct green flame (supposed to be due to the vapors of a Boric ether) constitutes the test for Boracic acid.

NOTE. It is to be observed that certain other substances may impart a green color to the flame. But, *First*, some of such substances are rare, viz.: compounds of Molybdenum, Tellurium, and Thallium. *Secondly*,

\* *M. W. Iles*, THE CHEMICAL NEWS for May 11, 1877, page 204.

Phosphoric acid gives a much feebler color than Boracic acid. *Thirdly*, Barium salts, if present, will be changed, by addition of Sulphuric acid, to Baric sulphate. This latter salt does not color the flame. *Fourthly*, Copper salts, if present, must be treated with Sulphuretted-hydrogen, and all Copper removed by filtration—before applying the test.)

### TARTRATES AND CITRATES.

**174.** Tartaric acid and Citric acid belong to the Group of organic acids, and their tests are described under that heading. (Page 84, *et seq.*)

### 175. Common Forms of Fourth Group Acids.

Oil of vitriol, Many sulphates.	Concentrated $\text{H}_2 \text{S O}_4$ .
Oxalic acid,	Crystals of $(\text{H}_2 \text{C}_2 \text{O}_4 + 2 \text{H}_2 \text{O})$ .
Bone-ash,	Mainly $\text{Ca}_3 (\text{P O}_4)_2$ .
Bi-chrome,	$\text{K}_2 \text{Cr}_2 \text{O}_7$ .
Borax, crystallized,	$(\text{Na}_2 \text{B}_4 \text{O}_7 + 10 \text{H}_2 \text{O})$ .



## CHAPTER XI.

### FIFTH GROUP OF ACIDS.

#### *THE MINERAL ACIDS.*

**176.** THIS Group includes two acids which are oftenest found in the insoluble form and in minerals.

They are—

Silicic acid,	$H_2 Si O_3.$
Fluohydric acid,	$H Fl.$

#### *SILICIC ACID ( $H_2 Si O_3$ ), OR SILICATES.*

**177.** Silicates that are soluble in water are of comparatively rare occurrence.

Silicates that are insoluble in water are very numerous. They are represented by a very large number of mineral substances, occurring in nature.

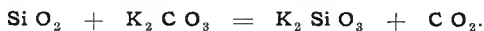
In general, silicates of the alkali metals (Potassium and Sodium) are soluble when the alkali metal predominates.

Silicic anhydride ( $SiO_2$ ), which is insoluble in water and acids (except Fluohydric), is also of very common occurrence in nature, in the form of rock-crystal, quartz, flint, etc.

#### *First Case.—Insoluble Silicates.*

**178.** Reduce the substance to as fine a powder as possible. Fuse a very small portion, on charcoal, with about four times its weight of a mixture of Potassic car-

bonate and Sodic carbonate. (The mixture fuses at a lower temperature than either salt singly.) New compounds are thus found that are soluble in water—viz., Sodic silicate and Potassic silicate, having the alkali metals in excess. (When it is certain that no easily reducible metals, like Lead, Silver, Copper, etc., which injure Platinum, are present, the fusion is best performed in a Platinum dish or on Platinum foil.) In any case, the fusion must be thoroughly performed.

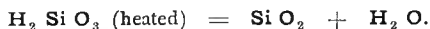


**179.** Pulverize the fused mass, and dissolve so far as possible by boiling in water. Filter, and to the filtrate add Chlorohydric acid and boil again. This should liberate Silicic acid, thus:



When the Silicic acid is in sufficient quantity, it appears as a thick jelly; in smaller quantity, it forms a gelatinous precipitate; in still smaller quantity, it may remain in solution.

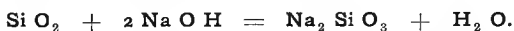
**180.** In any case, evaporate carefully, but completely, to dryness, preferably over a water-bath. In the process of concentration, the gelatinous precipitate should appear—if it has not been observed before that stage. Finally, heat the dry residue, to produce decomposition of Silicic acid, thus:



The Silicic anhydride ( $\text{SiO}_2$ ), thus produced, is insoluble. To it, add water and a few drops of Chlorohydric acid to dissolve soluble matters.

The presence, at this stage, of an insoluble residue, and one that feels gritty under a glass rod, indicates Silicic anhydride.

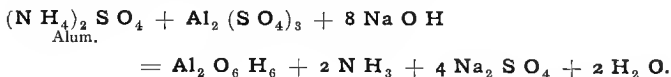
**181.** To confirm the test, pour off the soluble matters, and wash the residue with a little hot water. Then, to this insoluble part, add Sodic hydrate, and boil for a few minutes. This will convert the Silicic anhydride into soluble Sodic silicate ( $\text{Na}_2\text{SiO}_3$ ).



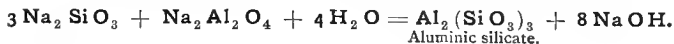
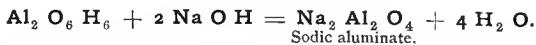
Pour some of this solution into a solution of Sodic aluminate (prepared by adding Sodic hydrate to a solution of Alum until the precipitate first formed is redissolved). A white, flocculent precipitate of Aluminic silicate shows the presence of a silicate.

### *Preparation and Use of Sodic Aluminate.*

#### FIRST STAGE.



#### SECOND STAGE.



### *Second Case.—Soluble Silicates.*

**182.** Soluble silicates are treated like insoluble silicates, except that the process described in paragraph 178 is omitted.



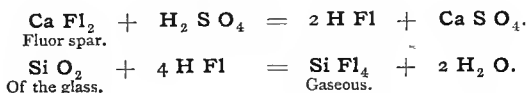
**FLUOHYDRIC ACID (HFl), or FLUORIDES.**

**183.** This acid is gaseous, and has the characteristic property of etching glass and porcelain. It does so by combining with the silicon of the glass or porcelain to form a gas, Fluoride of silicon ( $\text{SiFl}_4$ ), which escapes into the air.

The test is applied as follows. Prepare a slip of glass by melting a very thin layer of beeswax upon it, and then, when the beeswax coating has hardened by cooling, scratch some design, through the wax, with a pin.

Reduce the substance (if it is a solid) to a very fine powder. Place it in a lead dish. Add enough concentrated Sulphuric acid to the powder to make a paste. Cover the dish with the slip of glass, placing the sketch downward. Subject the whole to a gentle heat, but carefully avoid melting the lead or the wax.

After a sufficient time (varying, according to the conditions, from one hour to twenty-four), the wax being removed by melting it off, the glass will be found to be etched or engraved where the Fluohydric acid attacked it.

*Examples of Reactions.***184. Common Forms of Fifth Group Acids.**

Sand, flint, quartz, rock-crystal, etc.,	mainly, $\text{Si O}_2$ .
Very many mineral silicates.	
Silicate of soda (called soluble glass),	possibly, $\text{Na}_2 \text{Si O}_3 + \text{Aq.}$
Fluor-spar, Calcic fluoride,	$\text{Ca Fl}_2$ .

## CHAPTER XII.

### SIXTH GROUP OF ACIDS.

#### *THE ORGANIC ACIDS.*

**185.** ORGANIC ACIDS are those which are derived, directly or indirectly, naturally or artificially, from certain animal and vegetable matters.

More strictly defined, organic acids are those whose molecules contain one or more atoms of Carbon, *directly united* either with more Carbon or with Nitrogen or with Hydrogen.

**186.** These acids accord with the general formula for ternary acids—viz.,  $H-D-\bar{R}$ . H represents one, two, three, or four atoms of Hydrogen. D represents the linking dyad, usually Oxygen; and it must be remembered that the number of Oxygen atoms is usually the same as that of the one, two, three, or four atoms of positive Hydrogen.  $\bar{R}$  represents an electro-negative organic radicle, oftenest composed of Carbon, Oxygen, and Hydrogen.

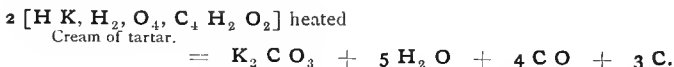
As an example, consider Tartaric acid,  $H_4O_4(C_4H_2O_2)$  (discussed on pages 21 and 86).

**187.** When an organic acid, or one of its metallic salts, is subjected to a low red heat, it is decomposed; a part of the Carbon is oxidized, and either escapes as Carbonic anhydride ( $CO_2$ ), or remains in the form of a Carbonate of the metal of the original salt. Another part of the Carbon

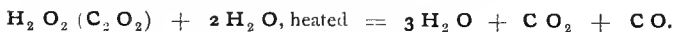
separates and remains as a black deposit of finely-divided charcoal. Thus :



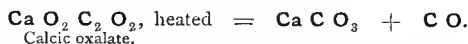
and



188. Oxalic acid is an exception to the foregoing statement. Its formula, when crystallized, is  $\text{H}_2\text{O}_2(\text{C}_2\text{O}_2) + 2 \text{H}_2\text{O}$ . When it is heated, it decomposes into water and two gaseous compounds of Carbon—Carbonic anhydride ( $\text{CO}_2$ ) and Carbonous oxide ( $\text{CO}$ ). Thus :



Oxalates are also exceptional, thus :



In these cases, as no excess of Carbon is left, of course no blackening occurs. The tests for this acid are described, therefore, with the inorganic acids of the Fourth Group of Acids. (See pp. 74-75.)

189. The organic acids are exceedingly numerous, being counted by hundreds. Not only are different vegetables characterized by the production of peculiar acids, but these, by the influences of growth, of decay, and of chemical agents, are easily modified into yet others.

Only a few of the most commonly-occurring organic acids will be described here.

### *The General Group Test.*

190. Heat carefully, on a fragment of porcelain, a portion of the substance to be tested. If charring occurs, it

indicates the probable presence of some member of this Group. (But the mere blackening of certain metallic salts must not be mistaken for charring. Thus, Copper salts turn black with heat, because of the formation of Cupric oxide,  $\text{CuO}$ ).

Next, proceed to make the individual tests, somewhat as follows.

### **ACETIC ACID, $\text{HO}(\text{C}_2\text{H}_3\text{O})$ , OR ACETATES.**

**191.** Acetic acid is the acid of *vinegar*. It is formed in excessive fermentation of wines, by oxidation of the Ethylic alcohol. It is now cheaply prepared by the distillation of wood, in close retorts.

Acetic acid, or its representative, may be tested for by adding to the solution in question, *concentrated* Sulphuric acid and a little Ethylic alcohol.

*Acetic ether* is thus formed; it may be recognized by its characteristic and agreeable odor—that of old cider or of cider vinegar.

Acetic ether is a substitution compound, after the water type, formed by the action of Acetic acid upon Ethylic alcohol.

### **Illustrative Formulas.**

Ethylic alcohol,	$\text{C}_2\text{H}_5 - \text{O} - \text{H}$ .
Acetic acid,	$\text{H} - \text{O} - \text{C}_2\text{H}_3\text{O}$ .
Acetic ether,	$\text{C}_2\text{H}_5 - \text{O} - \text{C}_2\text{H}_3\text{O}$ .
Ethylic ether,	$\text{C}_2\text{H}_5 - \text{O} - \text{C}_2\text{H}_5$ .

**TARTARIC ACID,  $\text{H}_4\text{O}_4\bar{\text{T}}$ , or  $\left. \begin{matrix} \text{H}_2 = \text{O}_2 = \\ \text{H}_2 = \text{O}_2 = \end{matrix} \right\} \text{C}_4\text{H}_2\text{O}_2$ .**

**192.** Tartaric acid is derived from the juice of the grape, where it exists as Potassio-calcic tartrate. During

the fermentation of wine, this double salt separates, and forms a deposit on the sides of the barrels. From this *crude Tartar* or *Argols*, as the deposit is called, Tartaric acid is afterward prepared.

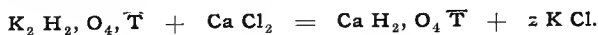
Tartaric acid is tetratomic, but bibasic. By this we mean that, while it has four atoms of positive Hydrogen, only two of them are replaceable by metals or by positive radicles. This peculiarity is illustrated on page 21.

### A.—Odor Test.

**193.** Heat a small portion of the dry substance, on porcelain. Attentively observe the odor evolved. Tartaric acid and Tartrates give a very characteristic, agreeable odor. The judgment is aided by heating a fragment of known Tartaric acid for comparison.

### B.—Calcium Chloride Test.

**194.** Make a neutral solution of the substance to be tested—or one that is feebly alkaline. To it, add solution of Calcic chloride. A white precipitate of Calcic tartrate ( $\text{CaH}_2\text{O}_4\bar{\text{T}}$ ) should appear. This precipitate should be soluble in Acetic acid. (Oxalates, under similar conditions, give a white precipitate of Calcic oxalate, which is *not* soluble in Acetic acid.)



Moreover, Calcic tartrate is soluble in cold solution of Sodid hydrate. If, upon trial with Sodid hydrate, a residue is left, filter; boil the clear filtrate; the appearance of a precipitate, which re-dissolves upon cooling, confirms the test.

### C.—Fenton's Test.\*

NOTE. Prepare a fresh solution of Sodic hypochlorite, by adding some solution of Bleaching-powder to solution of Sodic carbonate. Stir the whole, and filter. Use the filtrate.

**195.** To the solution, supposed to contain Tartaric acid or a Tartrate, add—

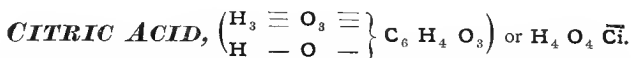
*First, a few drops* of a dilute solution of Ferrous sulphate. (Ferric solutions are inadmissible.)

*Secondly, a single drop* of very dilute Chlorine water or of solution of Sodic hypochlorite.

*Thirdly, add, very cautiously,* solution of Sodic hydrate.

If the test has been properly performed, and Tartaric acid is present, a fine violet coloration (supposed to be due to formation of Potassic or Sodic ferrate) should appear.

NOTE. It is a matter of some difficulty to hit the proper conditions for applying this test. These should be learned, by always making a trial with known Tartaric acid or Tartrate, before working with the problematical substance.



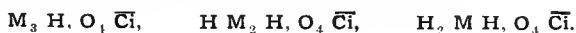
**196.** Citric acid occurs in lemons, limes, and many other fruits.

It is tetratomic, but tribasic. By this we mean that, although it contains four atoms of positive Hydrogen, only three of them are replaceable by metals or by positive radicles. This peculiarity gives rise to a variety of

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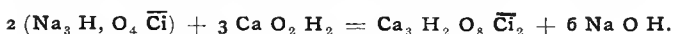
\* H. J. H. Fenton, CHEMICAL NEWS, 1876, Vol. 33, p. 190.

forms of salts. Three examples are given below, in which M represents one atom of a monad metal.



### *Lime-water Test.*

197. Add some of the solution to be examined, to a test-tube nearly full of Lime-water. In the cold, Citric acid should give no precipitate. (But Tartaric acid, Oxalic acid, Carbonic acid, and others, should give precipitates.) If, therefore, a precipitate is formed, filter. Now, boil the clear filtrate. A white precipitate indicates probable presence of Citric acid.



The Calcic citrite is soluble in Acetic acid.

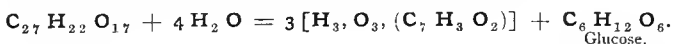
### **OXALIC ACID, $H_2 O_2 (C_2 O_2)$ , or $H_2 O_2 \bar{O}$ .**

198. This acid occurs as Potassic oxalate in sorrel. It also occurs in the rhubarb or pie-plant. Of late, it has been largely manufactured by the artificial decomposition of certain other organic matters: thus, it is prepared by the oxidation of starch or sugar by Nitric acid; also, by the action of Caustic potash upon saw-dust.

For the tests, see pp. 74-75-91.

### **TANNIN OR GALLO-TANNIC ACID ( $C_{27} H_{22} O_{17}$ ), AND GALLIC ACID, $H_3 O_3 (C_7 H_3 O_2)$ .**

199. These substances are usually found associated. In those cases where Tannin exists alone it easily changes, by aid of acids or fermentation, to Gallic acid.



Tannin exists in many vegetable substances, such as oak-bark, fustic, tea, etc. One of its principal sources is nut-galls. These are excrescences which grow on oak-leaves after they have been punctured by a certain insect.

Gallic acid exists, already formed, in a few substances; but it is usually prepared by the transformation of the Tannin contained in nut-galls.

Both Tannin and Gallic acid possess the property of producing, with Ferric salts, black or bluish precipitates (writing-inks).

**200.** Tannin is distinguished from Gallic acid as follows. To the solution, slightly acidulated by HCl, add a water-solution of gelatine (or glue). Tannin produces a curdy, white precipitate, sometimes called Tannate of gelatine. Under like circumstances, Gallic acid produces no precipitate.





## CHAPTER XIII.

### EXCEPTIONAL OXALATES, BORATES, AND PHOSPHATES.

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#### *Statement of the Case.*

**201.** THE following substances are exceptional, so far as their qualitative detection by the *ordinary* process is concerned, viz. :

Oxalates	}	of	}	Barium.
Borates				Strontium.
Phosphates				Calcium.
				Magnesium.

These substances are insoluble in water; they are soluble in Chlorohydric acid; they are insoluble in Ammonic hydrate.

**202.** If, now, one of these substances is given the chemist as a problem, he proceeds to test for the metal.

He finds that the substance dissolves in Chlorohydric acid, and he decides that the First Group metals are absent.

He adds Sulphuretted hydrogen, and decides that the Second Group metals are absent.

He adds Ammonic hydrate, etc., and obtains a white or gray precipitate, and *decides incorrectly that some metal or metals of the Third Group are present.*

Upon testing the filtrate for the Fourth Group metals, no precipitate is obtained, because the salt under examination has been thrown down, as if a member of the Third Group. An additional error is thus incurred, because members of the *Fourth Group are now erroneously decided to be absent*. Magnesium of the Fifth Group is missed in the same way.

### *When to Search for these Salts.*

**203.** *First*, when the original substance is insoluble in water; but is soluble in Chlorohydric acid, from which it is precipitable by Ammonic hydrate.

*Secondly*, when, in testing for the metals of the Third Group, an abundant precipitate of a white or gray color is produced, upon addition of Ammonic hydrate, although subsequent examination fails to reveal members of the Third Group.

*Thirdly*, when *ordinary* tests fail to show presence of either Barium, Strontium, Calcium, or Magnesium, or all of them, while preliminary examination—with the spectroscope or otherwise—leads the chemist to suspect the presence of one or more of them.

*Fourthly*, when, in testing for acids, either Oxalic, Boracic, or Phosphoric acid is detected, and when, at the same time, other conditions, like those just stated, do not render inadmissible the supposition of the presence of one or more of the salts in question.

### *Course of Treatment.*

#### *Test for an Oxalate.*

**204.** Carefully conduct the test as described at pp. 74-75.

If an Oxalate is detected, take a portion of the original material and change it into a Carbonate by ignition upon porcelain. Dissolve the Carbonate in Chlorohydric acid. There is thus produced a Chloride of the original metal; now proceed to test this solution for the metal, by the ordinary process.

*Test for a Phosphate.*

**205.** Carefully conduct the test as described at pp. 76-77.

If a Phosphate is detected, dissolve a portion of the original material in Chlorohydric acid, neutralize the solution with Ammonic acetate (see Phosphoric acid, p. 76), then add Ferric chloride carefully until all of the original Phosphate has been changed into Ferric phosphate,  $\text{Fe}_2(\text{PO}_4)_2$ . Filter, and, of course, the filtrate will contain the metal of the original salt, in the form of a Chloride. Test this solution for the metal according to the ordinary process.

*Test for a Borate.*

**206.** Carefully conduct the test as described on p. 78.

If a Borate is detected, dissolve some of the original salt in Chlorohydric acid, and add Sulphuric acid and Ethylic alcohol. The Sulphuric acid forms Sulphates of the metals, while the Boracic acid is liberated. Baric, Strontic, and Calcic sulphates are insoluble, the insolubility of the latter two being increased by presence of the alcohol. Filter, and test the insoluble Sulphates as follows. Boil them, for five minutes, in a mixture of Sodid carbonate and Potassic sulphate (see p. 44). This will change Calcic and Strontic sulphates to Calcic and Strontic carbonates. Baric sulphate remains unchanged. Filter, and thoroughly wash the precipitate with hot

water, and remove to a test-tube the precipitate, which should consist of one or all of the following substances, *and nothing else* :

Strontic carbonate,	$\text{Sr C O}_3$
Calcic carbonate,	$\text{Ca C O}_3$
Baric sulphate,	$\text{Ba S O}_4$

207. Now, warm this precipitate in *dilute* pure Chlorohydric acid. This will dissolve the Strontium and Calcium salts, and leave Baric sulphate undissolved.

Filter, and test the filtrate for Strontium and Calcium, exactly as prescribed at page 44, *et seq.*

The precipitate, supposed to consist of Baric sulphate, must be tested as described under the head of substances *insoluble in water and acids*. (Page 96.)

#### *The Course of Treatment for Magnesian Borate.*

208. For *Magnesian borate* a treatment different from the above is necessary, because Magnesian sulphate is soluble in dilute alcohol.

After adding Sulphuric acid (as described in paragraph 206), and filtering to collect the Baric, Strontic, and Calcic sulphates, the Magnesian sulphate will be found in the filtrate.

If we can be certain that, in a given case, the Boracic acid has all separated as a crystalline precipitate, with the Baric, Strontic, and Calcic sulphates, then we may proceed as follows.

To the filtrate, add Sodid hydrate; a precipitate of Magnesian hydrate ( $\text{MgO}_2\text{H}_2$ ) will appear, especially upon boiling. Filter again. Dissolve the precipitate in Chlorohydric acid (HCl), add Ammonic chloride ( $\text{NH}_4\text{Cl}$ ),

and Ammonic hydrate  $(\text{NH}_4)\text{HO}$ . The solution must be filtered, if it is not clear. To the clear part, add Hydro-di-sodic phosphate. The white crystalline precipitate of Ammonio-magnestic phosphate  $(\text{NH}_4\text{MgPO}_4)$  indicates Magnesium. (See page 48.)

NOTE. If a problematical substance is supposed to be or to contain Magnestic borate, it may well be examined in accordance with Chapter XIV. This Borate is not insoluble in acids, but the process, described in that chapter, is a good one for *separating the metal and acid*, and, hence, for overcoming the difficulty in the way of the ordinary process for the detection of the metal.



## CHAPTER XIV.

### TREATMENT OF INORGANIC SUBSTANCES, INSOLUBLE IN WATER AND ACIDS AND ORDINARY REAGENTS (EXCEPT FLUO- HYDRIC ACID).

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**209.** THE substances classed as insolubles are mostly natural minerals, and these are, principally, Silicates. There are a few artificial substances that are insoluble, as, for example, Baric sulphate. Many substances, however, which are insoluble in ordinary acids are soluble in certain special solvents. Thus, Plumbic sulphate is soluble in Ammonic acetate; Plumbic chromate is soluble in Sodid hydrate; Sulphur is soluble in Carbon di-sulphide.

When, therefore, a substance which is under examination appears to be insoluble in ordinary reagents, careful attention must be given to the preliminary tests; these often afford a suggestion which will readily lead to the discovery desired. (See page 99.)

#### *Process for Insolubles.*

**210.** Reduce the substance to a very fine powder; mix a small quantity of it with three times its weight of  $K_2CO_3$ , and *thoroughly* fuse the mixture, on charcoal, before the blow-pipe. Carefully examine the fused mass for the purpose of detecting any metallic globules. It is important not to err on this point.

If, by mistake, a salt of an easily-reducible metal is fused on *Platinum*, the latter may be permanently damaged. Even minute globules of reduced base-metal may form a readily-fusible alloy with *Platinum*, and produce discolorations and holes in the *Platinum*.

A. *No metallic globules appear.* Take a new and somewhat larger portion of the mixture of substance and flux, and fuse on *Platinum* foil or in a *Platinum* crucible. A reaction now takes place, by which the metal of the original salt changes place with the metal of the flux, thus :



**211.** The principles on which the process depends may be stated thus :

*First*, the original salt is changed into two salts—a *Carbonate*, of the unknown metal; a *Potassic or Sodic* salt, of the unknown acid.

*Secondly*, almost all *Carbonates* (except those of the *Alkali* metals) are insoluble in water.

*Thirdly*, almost all *Potassic* and *Sodic* salts are soluble in water.

**212.** Therefore, digest the fused mass in water, and filter. Test the filtrate for the *acid radicle* by the ordinary tests.

Test the precipitate for the *metal* by dissolving in *Chlorhydric* acid and proceeding as usual.

B. *Metallic globules appear.*

Continue the fusion on charcoal; remove the fused mass (a little charcoal does no harm), pulverize it, and digest it in water. Filter, and test the filtrate for the *acid radicle*.

Test the precipitate by dissolving the metallic globules in Nitric acid, if possible, and proceeding according to the directions for "Treatment of Metallic Alloys." (Page 53.)

NOTE. When a *Sulphate* is fused on charcoal, the salt is *reduced*, partially, at least, to a *Sulphide* by the deoxidizing power of the Carbon. Test for the Sulphide by placing a fragment of the fused mass upon a clean piece of Silver, and then adding a drop of water. (A silver coin may be cleaned by immersing it in Nitric acid, and then in water.) A black stain of Argentic sulphide ( $\text{Ag}_2\text{S}$ ) gives evidence of a *Sulphate* in the original substance.





## CHAPTER XV.

### PRELIMINARY EXAMINATION OF UNKNOWN SUBSTANCES.

---

**213.** THE preliminary tests are of great importance. They are valuable—

*As narrowing the field to be explored, by showing quickly that whole classes of substances are either absent, or are represented by the presence of some member or members of them;*

*As affording cumulative evidence in favor of a decision reached by later processes;*

*As teaching the student to observe carefully every phenomenon, and then to employ intelligently the information he obtains.*

#### *Course of Preliminary Examination.*

##### *The Substance is a Liquid.*

**214.** It may be fixed in one or more of the following classes :

*a.* Water.

*b.* Aqueous solutions of neutral organic bodies, as Cane-sugar.

*c.* Aqueous solutions of metallic salts of First, Second, Third, Fourth, or Fifth Group.

- d.* Aqueous solutions of  $K O H$ ,  $Na O H$ ,  $N H_4 O H$ —*i. e.*, the alkalies.
- e.* Aqueous solutions of  $H Cl$ ,  $H_2 S O_4$ ,  $H N O_3$ , and some other acids.
- f.* Ethylic alcohol (and some other alcohols).
- g.* Ethylic-alcoholic solutions. (Alcohol dissolves some metallic salts; many resins; organic bases; and essential oils.)
- h.* Ethylic ether and other ethers.
- i.* Ethereal solutions. (Ether is the appropriate solvent for fats.)
- k.* Fatty or essential oils.

215. Observe the **Odor**.

216. Observe the **Taste**. (*Beginners must omit this test.* More experienced chemists may apply to the tongue a minute drop of the liquid on the end of a small glass rod *which has a rounded point*; a *sharp* edge might be the means of introducing a poison into the circulation. The liquid should be thrown out, never swallowed.)

217. Observe the **Reaction**. Place a drop of the liquid on red litmus-paper and another on blue, and observe the change of color. Addition of a drop of distilled water sometimes favors this test.

218. Observe the **Residue from Evaporation**.

Carefully heat a drop or two of the liquid on a piece of glass, or on a stout glass rod, or on a Platinum foil; continue the heating until the liquid evaporates to dryness; gradually increase the heat, and observe its effect upon the residue (in case any residue remains).

Some liquids—as distilled water, for example—evaporate without leaving any residue, or showing any chemical phenomena.

Other liquids take fire, or they leave a residue, or the residue itself may burn.

NOTE. Liquid mercury and mercurial amalgams are usually at once recognized by inspection. They are tested like solid metals and alloys. (See page 53.)

### *The Substance is a Solid.*

**219.** It may be fixed in one of the following classes:

- l.* Metals and alloys.
- m.* Non-metals.
- n.* Solid acids or alkalies.
- o.* Salts.
- p.* Organic neutral bodies.

### *Heat a Fragment on Charcoal.*

**220.** Heat carefully before the blowpipe, on charcoal, a small fragment of the substance (about the size of a pin-head), and observe the effects of the heating.

Observe what coatings of oxide, if any, form upon the coal; whether near or distant; whether volatilizable by the blow-pipe flame or not.

When the assay-piece (if a metal) has cooled, endeavor to flatten it with a hammer on a clean anvil. This test may distinguish Lead, which is malleable, from Bismuth, which is brittle.

**Table of Phenomena observed upon Heating  
Certain Substances on Charcoal.**

<i>Name of Substance.</i>	<i>The Assay-Piece.</i>	<i>The Coatings on the Coal.</i>
Silver,	fuses to a globule, which remains bright; the globule sometimes "sprouts" on cooling.	
Lead,	fuses readily and remains fused for a long time; quickly tarnishes (by oxidizing),	dark yellow when hot, light yellow when cold.
Mercury,	boils and vaporizes.	
Arsenic,	volatilizes rapidly; peculiar garlic odor to the fumes,	white; easily driven away.
Antimony,	fuses readily to a globule, which takes fire; if dropped on the table, the fragments burn; globule, <i>brittle</i> ,	white; near the assay-piece.
Tin,	fuses readily,	luminous when hot; white when cold.
Gold,	fuses with difficulty,	none.
Platinum,	infusible; readily fuses upon addition of a little Lead,	none.
Copper,	very difficult to fuse.	
Bismuth,	fuses readily; globule, <i>brittle</i> ,	dark yellow when hot; light yellow when cold.
Cadmium,	takes fire; emits brown fumes,	brown.
Nickel,	infusible.	
Iron,	infusible.	
Zinc,	fuses readily; burns with a greenish flame and thick white smoke,	yellow white hot; white when cold; non-volatile.
Aluminum,	fusible in small portions.	
Magnesium,	the entire piece burns with dazzling light; leaves a mass of white oxide,	white; non-volatile.
Potassium,	takes fire; burns with a violet flame.	
Sodium,	takes fire; burns with an orange flame.	
Nitrates and Chlorates,	deflagrate; burn the charcoal.	

**Heat a Fragment in a Tube closed at one End.**

**221.** Observe any change that may result as described in the following table :

<i>Name of Substance.</i>	<i>The Assay-Piece.</i>	<i>The Sublimate.</i>
Iodine,	volatilizes completely; with violet fumes; and choking odor,	black crystals.
Sulphur,	turns dark-brown; then becomes yellow again by more heating or by cooling. When the amount is small, it volatilizes completely,	white or yellow.
Many sulphides,	turn yellow, brown, or black.	yellow or brown.
Mercury salts,	often volatilize completely,	varying from white to black.
Arsenic compounds,	often volatilize completely,	white crystals, of $\text{As}_2\text{O}_3$ . black, of <b>As</b> . red or yellow [ $\text{As}_2\text{S}_3$ , etc.].
Copper salts,	often give black, <b>Cu O</b> .	
Antimony compounds,	some are volatile; some are fixed,	may afford white needle-shaped crystals.
Ammonium compounds,	volatilize completely,	white; may be removed and tested for $\text{NH}_3$ by Nessler's test. (Page 50.)

Organic compounds,	puff up; charr, and blacken,	smoky fumes and tarry products.
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### *Try the Flame Color Tests.*

222. Reduce a portion of the salt to a very fine powder; take a Platinum wire and clean it (by rubbing it with a little wet sand, and then washing it with water and Chlorohydric acid, and then heating it in the lamp-flame); moisten the wire with Chlorohydric acid, and then dip it in the powder under examination. Now, hold the wire and the powder in the edge of the Bunsen flame, or in the inner flame of the blow-pipe; observe the color of the outer flame.

<i>Name of Substance.</i>	<i>Color of the Outer Flame.</i>
Copper compounds,	blue; changing to green when the $\text{Cu Cl}_2$ has burned off.
Barium compounds (the sulphate with difficulty),	yellowish-green.
Strontium compounds,	crimson.
Calcium compounds,	orange-red.
Potassium compounds,	violet.
Sodium compounds,	orange-yellow.
Lithium compounds,	carmine,
Boracic acid,	green.
Phosphoric acid,	bluish-green.

### *Try the Tests with the Borax Bead.*

223. Roast the substance on charcoal, or, better, in an open tube; but avoid fusion of the substance. The roasting *expels volatile substances*; it also *turns metals*

into oxides, in which latter condition they are readily dissolved by Borax. Make a clear bead of Borax on a Platinum wire, and into it fuse a small quantity of the roasted substance.

If too much of the substance has been introduced, break the bead, and fuse one or two of its fragments into a fresh Borax bead.

The *outer flame* is oxidizing, because there the assay-piece is heated in contact with abundance of atmospheric oxygen; the *inner flame* is reducing, because there the substance is highly heated in contact with the mass of reducing—*i. e.*, de-oxidizing—coal-gas.

<i>Name of Substance.</i>	<i>In the Oxidizing Flame.</i>	<i>In the Reducing Flame.</i>
Copper,	green, when hot; blue, when cold;	opaque and reddish from formation of $\text{Cu}_2\text{O}$ .
Cobalt,	intense blue or blue-black;	intense blue.
Nickel,	violet, when hot; brown, when cold;	gray and cloudy.
Ferric compounds,	yellow or red, when hot; lighter color, when cold;	bottle-green.
Manganic compounds,	intense reddish-violet;	almost colorless.
Chromic compounds,	yellow or reddish, when hot; yellowish green, when cold;	green.

### *Try the Influence of Solvents.*

**224.** There are three important rules to be observed in dissolving substances, preparatory to testing them :

(a) Reduce the substance to a very fine powder, before trying solvents.

(b) Use but a small quantity of the substance upon the first trial.

(c) Try the substance thoroughly with water, before using other solvents.

1st. Add some water to the substance in question, and gently warm the mixture. Increase the heat to boiling, if necessary. Apply a drop of the liquid to litmus-paper.

If it appears doubtful if anything has dissolved, filter a few drops of the mixture, and test the clear filtrate, by evaporating a drop of it upon a glass rod. If a residue is left, it indicates that something has dissolved.

With few exceptions, hot water dissolves all salts of Potassium, Sodium, and Ammonium; all Nitrates; all Chlorides (except  $\text{AgCl}$  and  $\text{Hg}_2\text{Cl}_2$ ).

2d. If water fails to dissolve the substance, try *dilute Chlorohydric acid*. If this fails, try concentrated Chlorohydric acid.

In using acids as solvents, always try the *dilute* acid first; many substances, which are easily soluble in certain dilute acids, are nearly insoluble in the same acids when they are concentrated.

3d. If the foregoing trials are ineffectual, take a fresh portion of the original substance; try *dilute Nitric acid*. As a next resort, try *concentrated Nitric acid*.

4th. Next try, upon a fresh portion, *Aqua-regia* (a mixture of two measures of Chlorohydric acid to one of Nitric acid).

5th. Next try a small portion of the original substance with *Ammonic hydrate*.



6th. Next try a small portion with *Sodic hydrate*.

7th. Next try a small portion with Ethyl alcohol ( $C_2H_5OH$ ). Take care not to set the alcohol on fire at the lamp.

8th. Other special solvents, like Ethyl ether, Chloroform, Glycerine, Carbon di-sulphide, etc., may be used in examination of substances, which, by their previous preliminary examination, seem to promise success by that treatment; but, in ordinary cases, they are quite uncalled for.





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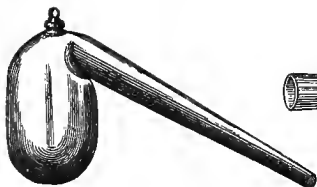
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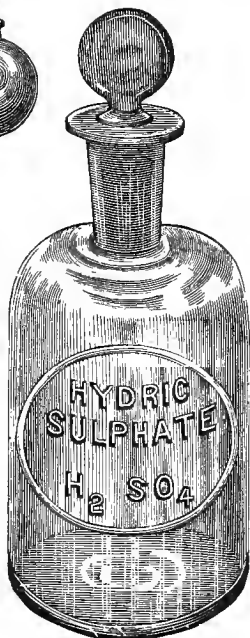


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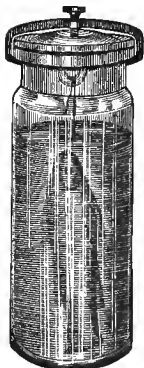
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