Inorganic Qualitative Analysis

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Analytical Chemistry

Inorganic Qualitative Analysis

QUALITATIVE ANALYSIS OF INORGANIC COMPOUNDS

Introduction

Inorganic compounds may be found in a liquid or in a solid state. If the material was found as liquid or in a solution, we have to follow many steps as the following:

- Notice the color, smell and other physical properties.
- Check the acidity and alkalinity using litmus paper or any other indicators to be sure if the compound is neutral, acidic salt, or basic salt.
- Evaporate small amount of the liquid to dryness. If any solid residues remained, the material may be salt o alkali hydroxides and earth elements which give brown PPt with $AgNO_3$, blue PPt with $CuSO_4$ or brown reddish PPt with $FeCl_3$.
- Acid radical (anions) or basic radical (cations) of the compound or both of them should be tested.

If the material was found as solid, we have to dissolve the solid material in an appropriate solvent to be salt solution.

Solubility tests:

Try to dissolve the solid material in the following solvents at the order of:

- Water, Hot water
- HCl_{dil}, HCl_{dil} + heating
- HCl_{conc}, HCl_{conc} + heating
- $HNO_{3\ dil}$, $HNO_{3\ dil}$ + heating
- $HNO_{3\ conc}$, $HNO_{3\ conc}$ + heatin

- Aqua regia (3ml of HCl_{conc} + 1ml of $HNO_{3\ conc}$)

Note: The solution of any concentrated acid should be diluted with water before any test.

Equilibrium-constant

Equilibrium-constant expressions are algebraic equations that relate the concentrations of reactants and products in a chemical reaction to one another by means of a numerical quantity called an equilibrium constant.

Consider the generalized equation for a chemical equilibrium

$$m\mathrm{M} + n\mathrm{N} \rightleftharpoons p\mathrm{P} + q\mathrm{Q}$$

where the capital letters represent the formulas of participating chemical species and the italic letters are the small integers required to balance the equation. Thus, the equation states that *m* moles of M react with *n* moles of N to form *p* moles of P and *q* moles of Q. The equilibrium-constant expression for this reaction is

$$K = \frac{[P]^p\ [Q]^q}{[M]^m\ [N]^n}$$

Where the letters in brackets represents the molar concentration of dissolved solutes.

Cations

Cations are divided into six groups on the basis of solubility product. Each group precipitated with certain reagent named as the group reagent.

Electrolytes are solutes which ionize in a solvent to produce an electrically conducting medium.

Strong electrolytes ionize completely whereas weak electrolytes are only partially ionized in the solvent.

The group reagent is an electrolyte which gives negative ion (anion). The concentration of this anion should be enough to reach the solubility product of its derivatives from certain metals.

Each salt has a certain solubility product at certain tempereature.
When an aqueous solution is saturated with a sparingly soluble salt, one or more equilibria will be established. With silver chloride, for example,

$$AgCl_{(s)} \rightleftharpoons AgCl_{(aq)}$$
$$AgCl_{(aq)} \rightleftharpoons Ag^+ + Cl^-$$

The solubility product constant (K_{sp}) of $AgCl_{(aq)}$ = $[Ag^+]\,[Cl^-]$
$[Ag^+]\,[Cl^-]$ in solution > K_{sp} of AgCl , the salt will precipitated.

So if the product $[Ag^+]\,[Cl^-]$ less than K_{sp} of AgCl, we can add excess of silver or chloride ions to the solution to increase the product $[Ag^+]\,[Cl^-]$ over K_{sp}

Classifications Of Cations

Group	Group reagent	Ions	Formula of precipitate	Distinguishing features
I **Silver group**	Dilute HCl	Ag^{+}, Pb^{+2}, Hg_2^{+2}	AgCl, $PbCl_2$, Hg_2Cl_2	Chlorides insoluble in cold dilute HCl
II **Copper and Arsenic group**	H_2S in presence of dilute HCl	Hg^{+2}, Pb^{+2}, Bi^{+3}, Cu^{+2}, Cd^{+2}, Sn^{+2}, As^{+3}, Sb^{+3}, Sn^{+4} , As^{+5}	HgS, PbS, Bi_2S_3, CuS, CdS, SnS, As_2S_3, Sb_2S_3, SnS_2 , As_2S_5	Sulfides insoluble in dilute HCl
III **Iron group**	NH_4OH in presence of NH_4Cl	Al^{+3}, Cr^{+3}, Fe^{+2}, Fe^{+3}	$Al(OH)_3$, $Cr(OH)_3$, $Fe(OH)_2$, $Fe(OH)_3$	Hydroxides PPt
IV **Zinc group**	H_2S in presence of NH_4OH, NH_4Cl	Ni^{+2}, Co^{+2}, Mn^{+2}, Zn^{+2}	NiS, CoS, MnS, ZnS	Sulfides PPt
V **Calcium group**	$(NH_4)_2CO_3$ in presence of NH_4OH, NH_4Cl	Ca^{+2}, Sr^{+2}, Ba^{+2}	$CaCO_3$, $SrCO_3$, $BaCO_3$	Carbonates PPt
VI **Alkali group**	No group reagent	Mg^{+2}, Na^{+}, K^{+}, NH_4^{+}	----------	----------

First group

Silver (Ag^{+}), Lead (Pb^{+2}), Mercury (Hg_2^{+2})

Simple salt test of silver

Silver, the white metal, is soluble in concentrated nitric acid. Its symbol is derived from the Latin name Argentum (Ag).

1. $AgNO_3 + HCl_{dil} \longrightarrow AgCl_{\text{white PPt}} + HNO_3$

   $AgCl + 2NH_3 \longrightarrow [Ag(NH_3)_2]Cl_{\text{Amino silver chloride}}$

   $AgCl_{\text{white PPt}}$ is soluble in excess of ammonia solution

2. $AgNO_3 + KI \longrightarrow AgI_{\text{Yellow PPt}} + KNO_3$

   $AgI_{\text{Yellow PPt}}$ is insoluble in ammonia solution

3. $AgNO_3 + KCN \longrightarrow AgCN_{\text{white PPt}} + KNO_3$

   $AgCN + KCN \longrightarrow K[Ag(CN)_2]_{\text{Argento pot. cyanide}}$

   $AgCN_{\text{white PPt}}$ is soluble in excess of KCN solution

4. $AgNO_3 + K_2CrO_4 \longrightarrow Ag_2CrO_{4\ \text{dark red PPt}} + 2KNO_3$

   $Ag_2CrO_{4\ \text{dark red PPt}}$ is soluble in HNO_3 and ammonia solution

5. $2AgNO_3 + 2NaOH \longrightarrow Ag_2O_{\text{Brown PPt}} + 2NaNO_3 + H_2O$

   $Ag_2O_{\text{Brown PPt}}$ is soluble in HNO_3 and ammonia solution

6. $2AgNO_3 + H_2S \longrightarrow Ag_2S_{\text{Black PPt}} + 2HNO_3 + H_2O$

   $Ag_2S_{\text{Black PPt}}$ is soluble in hot $HNO_{3\ dil}$

Simple salt test of Lead

Lead symbol is derived from the Latin name Plumbum (Pb).

1. $Pb(NO_3)_2 + HCl_{dil} \longrightarrow PbCl_{2\ white\ PPt} + HNO_3$

   $PbCl_{2\ white\ PPt}$ is soluble in hot water and return again after cooling.

2. $Pb(NO_3)_2 + 2KI \longrightarrow PbI_{2\ Yellow\ PPt} + 2KNO_3$

   $PbI_{2\ Yellow\ PPt}$ is slightly soluble in hot water.

3. $Pb(NO_3)_2 + K_2CrO_4 \longrightarrow PbCrO_{4\ Yellow\ PPt} + 2KNO_3$

   $PbCrO_{4\ Yellow\ PPt}$ is insoluble in acetic acid but soluble in HNO_3 and NaOH solution

4. $Pb(NO_3)_2 + H_2SO_{4\ dil} \longrightarrow PbSO_{4\ White\ PPt} + HNO_3$

   $PbSO_{4\ White\ PPt}$ is soluble in ammonium acetate and NaOH solution

5. $Pb(NO_3)_2 + H_2S \longrightarrow PbS_{Black\ PPt} + HNO_3$

   $PbS_{Black\ PPt}$ is soluble in hot $HNO_{3\ dil}$

6. $Pb(NO_3)_2 + NaOH \longrightarrow Pb(OH)_{2\ White\ PPt} + NaNO_3$

   $Pb(OH)_{2\ White\ PPt}$ is soluble in excess of NaOH

   $Pb(OH)_{2\ White\ PPt} + 2NaOH \longrightarrow Na_2PbO_2 + 2\ H_2O$

Simple salt test of Hg_2^{++}

Mercury is a bright liquid metal and its symbol is taken from the Latin name, Hydrarygrum. Its vapor is toxic

1. $Hg_2(NO_3)_2 + HCl_{dil} \longrightarrow Hg_2Cl_{2\ white\ PPt} + HNO_3$

   $Hg_2Cl_{2\ white\ PPt}$ is soluble in aqua regia (HCl_{conc} : $HNO_{3\ conc}$, 3:1).

   $Hg_2Cl_{2\ white\ PPt}$ changed to black PPt by adding ammonia solution.

   $Hg_2Cl_{2\ white\ PPt} + 2NH_3 \longrightarrow Hg_{black} + NH_4Cl +$

   $Hg(NH_2)Cl_{\ white\ PPt\ of\ amino\ mercuric\ chloride}$

2. $Hg_2(NO_3)_2 + 2KI \longrightarrow Hg_2I_{2\ green\ yellowish\ PPt} + 2KNO_3$

   $Hg_2I_{2\ green\ yellowish\ PPt}$ is soluble in excess of KI

   $Hg_2(NO_3)_2 + 2KI \longrightarrow K_2(HgI_4)_{\ mercuric\ pot.\ iodide} + Hg$

3. $Hg_2(NO_3)_2 + H_2S \longrightarrow HgS_{black\ PPt} + Hg_{Black} + 2HNO_3$

4. $Hg_2(NO_3)_2 + K_2CrO_4 \longrightarrow Hg_2CrO_{4\ Brown\ PPt} + 2KNO_3$

   $Hg_2CrO_{4\ Brown\ PPt}$ changed into red crystalline PPt by boiling.

5. $Hg_2(NO_3)_2 + 4NaOH \longrightarrow HgO + Hg_2O_{Black\ PPt} + Hg + 4NaNO_3 + 2H_2O$

6. $2Hg_2(NO_3)_2 + 4NH_3 + H_2O \longrightarrow 3NH_4NO_3 + 2Hg + HgO.Hg(NH_2)NO_3$

   Black PPt of Alkali amino mercuric nitrate

7. $2Hg_2(NO_3)_2 + SnCl_2 \longrightarrow Hg_2Cl_{2\ white\ PPt} + Sn(NO_3)_2$

$Hg_2Cl_{2\ white\ PPt} + SnCl_{2\ excess} \longrightarrow 2Hg_{black\ PPt} + SnCl_4$

Results

Reagent	Ag^+	Pb^{++}	Hg_2^{++}
HCl_{dil}	White PPt of AgCl which is soluble in ammonia		
KI		Yellow PPt of PbI_2 which is soluble in hot water.	
KCN			
K_2CrO_4			
NaOH			
H_2S			
NH_4OH			
$H_2SO_{4\ dil}$			
$SnCl_2$	------	------	White PPt of Hg_2Cl_2 which converted into black with excess of SnCl2

Mixture of the first group

The group will be precipitated according to the solubility product. Chlorides of the first group have the lowest solubility product comparing with the second, third,….or six group. So, the first group will be precipitated as chlorides while the chlorides of other groups will be soluble.

The group reagent of the first group is diluted HCl.
The precipitated ion of the first group is chloride ion.

Small amount of mixture in test tube + HCl_{dil} ⟶ white PPt

Put all mixture in a beaker + excess amount of HCl_{dil} and filter the mixture and collect the PPt

PPt ($PbCl_2$, AgCl, or Hg_2Cl_2)

Add hot water

Filtrate 1 — May be contains $PbCl_2$

Precipitate 1 — May be contains AgCl or Hg_2Cl_2 or both

Add excess of NH_4OH and filter

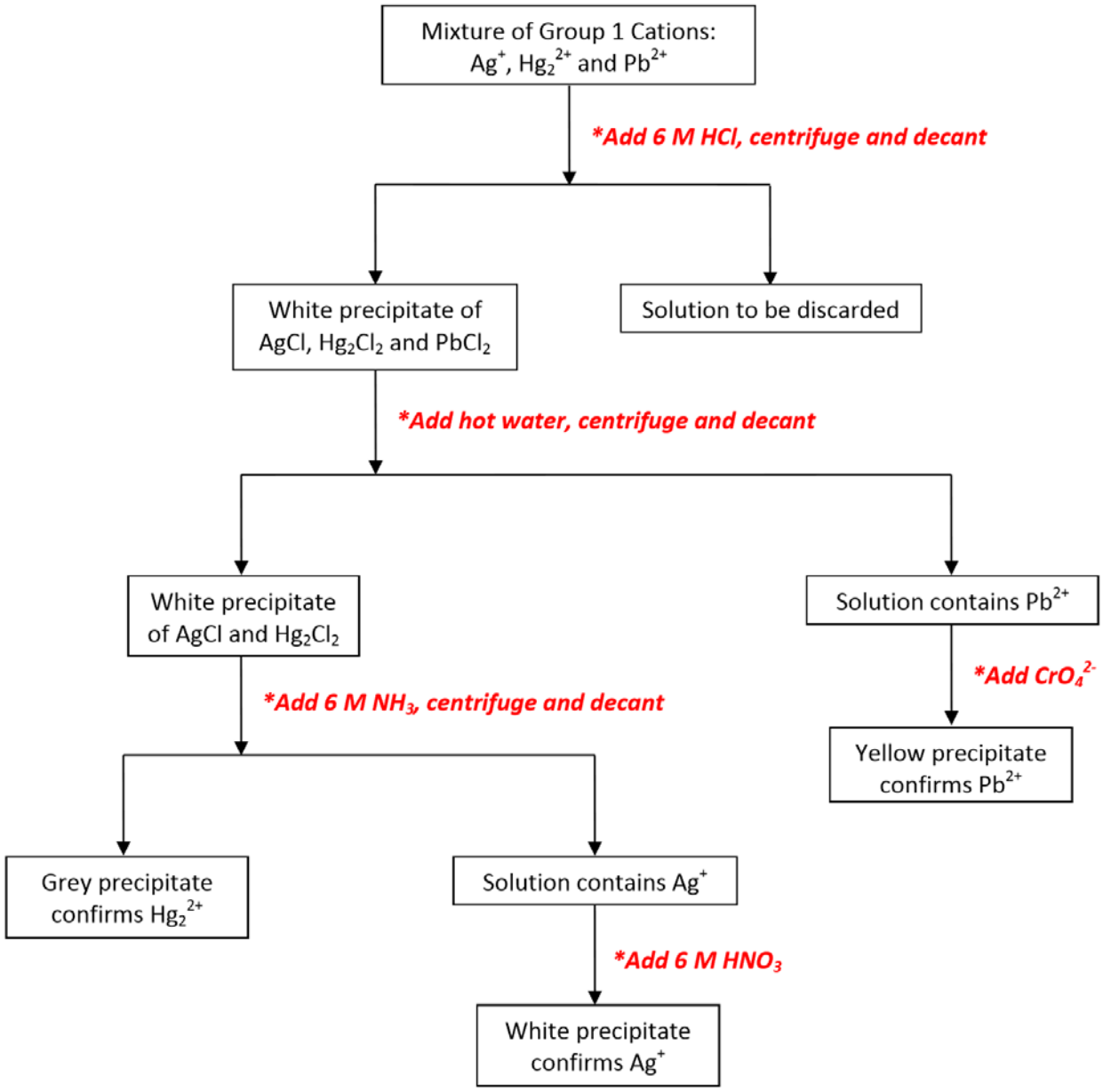
Filtrate 2 — May contains Ag

Add HNO_3, if White PPt, there is Ag

Precipitate 2 — Black PPt, Hg

Concerning Filtrate 1,

1. Small portion cooled under water stream, if white PPt, it is Pb
2. small portion + $H_2SO_{4\ dl}$, if white PPt, it is Pb
3. small portion + potassium chromate , if Yellow PPt, it is Pb


Mixture of Group 1 Cations: Ag^+, Hg_2^{2+} and Pb^{2+}
*Add 6 M HCl, centrifuge and decant
White precipitate of AgCl, Hg_2Cl_2 and $PbCl_2$
Solution to be discarded
*Add hot water, centrifuge and decant
White precipitate of AgCl and Hg_2Cl_2
Solution contains Pb^{2+}
*Add CrO_4^{2-}
Yellow precipitate confirms Pb^{2+}
*Add 6 M NH_3, centrifuge and decant
Grey precipitate confirms Hg_2^{2+}
Solution contains Ag^+
*Add 6 M HNO_3
White precipitate confirms Ag^+

Second group

Copper (Cu^{++}), Cadmium (Cd^{++}), Bismuth (Bi^{+3}), Mercury (Hg^{+2}), Antimony (Sb^{+3}), Arsenic (As^{+3}, As^{+5}), Tin (Sn^{+2}, Sn^{+4})

The second group is divided into two sub groups, G II-A and group II-B according the solubility of their sulfides in yellow ammonium sulfide where GII-A insoluble while GII-B is soluble.

Group II-A includes Copper (Cu^{++}), Cadmium (Cd^{++}), Bismuth (Bi^{+3}) and Mercury (Hg^{+2}).

Group II-B includes Antimony (Sb^{+3}), Arsenic (As^{+3}, As^{+5}) and Tin (Sn^{+2}, Sn^{+4})

Simple salt test of Hg_2^{++}

1. $3HgCl_2 + 2H_2S \longrightarrow Hg_3Cl_2S_2\ _{\text{white PPt}} + HNO_3$

   $Hg_3Cl_2S_2 + H_2S_{excess} \longrightarrow 2HCl + 3HgS\ _{\text{black PPt}}$

2. $HgCl_2 + 2KI \longrightarrow HgI_2\ _{\text{red PPt}} + 2KCl$

   $HgI_2\ _{\text{red PPt}}$ is soluble in excess of KI

   $HgI_2 + 2KI\ _{excess} \longrightarrow K_2(HgI_4)$

   mercuric pot. iodide

3. $HgCl_2 + NH_4OH \longrightarrow HgNH_2Cl + HCl + H_2O$

   White PPt of amino mercuric chloride

4. $HgCl_2 + NaOH \longrightarrow NaCl + HgOHCl\ _{\text{red PPt}}$

Simple salt test of Cu^{++}

Copper is a red metal and its symbol is taken from the Latin name Cuprum.

1. $CuSO_4 + H_2S \longrightarrow H_2SO_4 + CuS_{\text{Black ppt}}$

2. $2CuSO_4 + 2NH_4OH \rightarrow CuSO_4.Cu(OH)_{2\ \text{green PPt}} + (NH_4)_2SO_4$

   $CuSO_4.Cu(OH)_2 + 4NH_4OH_{excess} \longrightarrow 2Cu(NH_3)_4SO_4 + 8H_2O$

   Deep blue color of amino copper sulfate

3. $CuSO_4 + 2NaOH \longrightarrow Cu(OH)_{2\ \text{Blue greenish PPt}} + Na_2SO_4$

   $Cu(OH)_{2\ \text{Blue greenish PPt}} \xrightarrow{\Delta} CuO_{\text{Black ppt}} + H_2O$

4. $CuSO_4 + 2KI \longrightarrow CuI_{2\ \text{white PPt}} + K_2SO_4$

   $2CuI_{2\ \text{white PPt}} \longrightarrow Cu_2I_2 + I_{2\ \text{Brown color}}$

5. $CuSO_4 + 2KCN \longrightarrow Cu(CN)_{2\ \text{Brown yellowish PPt}} + K_2SO_4$

   $Cu(CN)_2 + 2KCN_{\text{excess}} \longrightarrow K_2[Cu(CN)_4]$

   Copprous pot. Cyanide

6. $CuSO_4 + 2KSCN \longrightarrow Cu(SCN)_{2\ \text{Black PPt}} + K_2SO_4$

   $Cu(SCN)_{2\ \text{Black PPt}}$ **is soluble in excess of KSCN giving a green color**

Simple salt test of Cd^{++}

1. $CdCl_2 + H_2S \longrightarrow CdS_{\text{Yellow ppt}} + 2\,HCl$

2. $CdCl_2 + 2NH_4OH \rightarrow Cd(OH)_{2\ \text{white PPt}} + 2NH_4Cl$

$Cd(OH)_{2\ \text{white PPt}} + NH_4OH_{\text{excess}} \longrightarrow [Cd(NH_3)_4]\,(OH)_2$

amino cadmium hydroxide

3. $CdCl_2 + 2NaOH \longrightarrow Cd(OH)_{2\ \text{white PPt}} + 2NaCl$

4. $CdCl_2 + 2KCN \longrightarrow Cd(CN)_{2\ \text{white PPt}} + 2KCl$

$Cd(CN)_2 + 2KCN_{\text{excess}} \longrightarrow K_2[Cd(CN)_4]$

Cadmium pot. Cyanide

5. $2\,CdCl_2 + K_4[Fe(CN)_6] \longrightarrow Cd_2[Fe(CN)_6] + 4\,KCl$

White PPt of
cadmium ferro cyanide

Simple salt test of Bi^{+3}

1. $BiCl_3 + 3H_2S \rightarrow Bi_2S_{3\ \text{Brown ppt}} + 6\,HCl$

2. $BiCl_3 + 3NH_4OH \rightarrow Bi(OH)_{3\ \text{white PPt}} + 3NH_4Cl$

3. $3\,BiCl_3 + 3H_2O \rightarrow 3(BiO)Cl_{\ \text{white PPt}}$

4. $2\,BiCl_3 + 3Na_2SnO_2 + 6\,NaOH \rightarrow$

$2Bi + 3Na_2SnO_3 + 6NaCl + 3H_2O$

<u>Preparation of Na_2SnO_2 :</u>

$SnCl_2 + 2\,NaOH \rightarrow Sn(OH)_{2\ \text{White ppt}} + 2\,NaCl$

$Sn(OH)_2 + 2NaOH_{\text{excess}} \rightarrow Na_2SnO_2 + 2\,H_2O$

Results

Reagent	Hg^{+2}	Cu^{+2}	Cd^{+2}	Bi^{+3}
H_2S	White PPt of $Hg_3Cl_2S_2$ which converted into brown and then black in excess of H_2S			
KI				
NH_4OH				
NaOH				
$SnCl_2$				
KCN				
KSCN				
K_4 $[Fe(CN)_6]$				
H_2O				
Na_2SnO_2				

Mixture of the second group

The Mixture of the second group

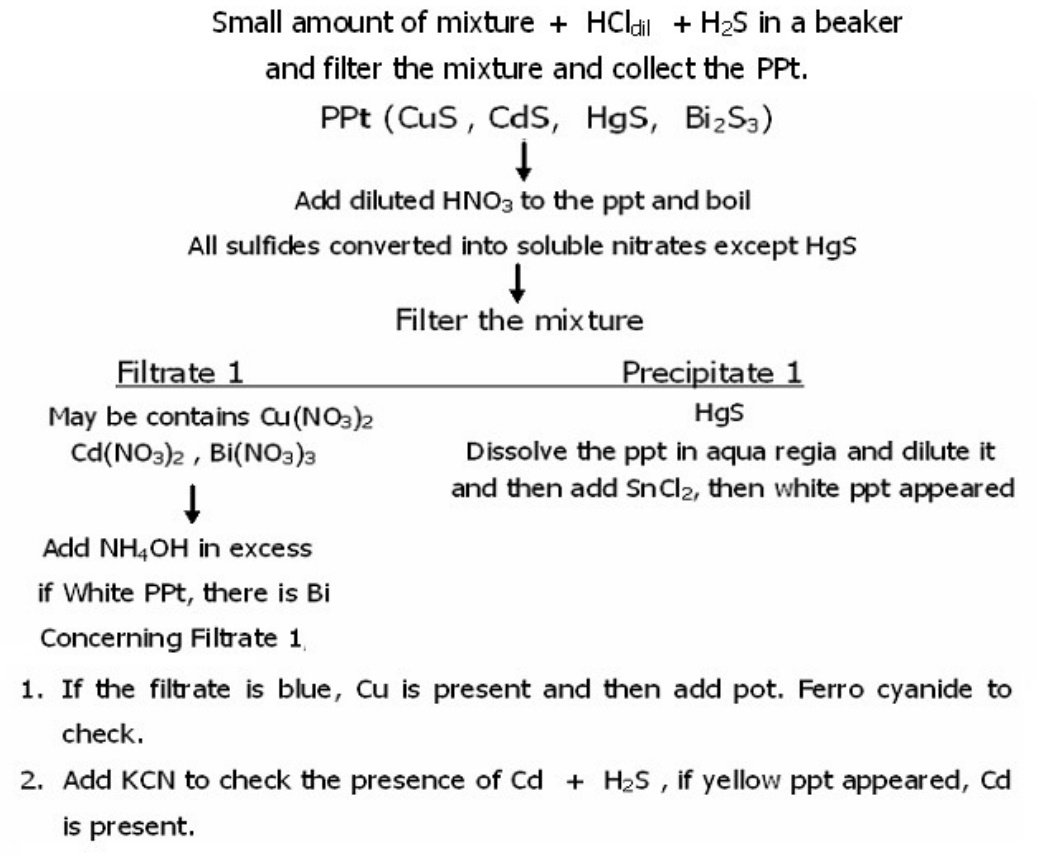
The group will be precipitated according to the solubility product. Sulfides of the second and fourth groups have the lowest solubility product comparing with the third,….or six group. So, the second and fourth groups will be precipitated as sulfides while the sulfides of other groups will be soluble.

The solubility products of the second group sulfides is less than the solubility products of the fourth group sulfides. Therefore we have to decrease the concentration of sulfide ion using the common effect by using HCl_{dil} and then add H_2S.

The group reagent of the second group is hydrogen sulfide (H_2S).

The precipitation ion of the second group is sulfde ion.

The mixture should acidified first using HCl_{dil} (0.3M) as a controlling precipitation ion.



Concerning Filtrate 1,

1. If the filtrate is blue, Cu is present and then add pot. Ferro cyanide to check.
2. Add KCN to check the presence of Cd + H_2S , if yellow ppt appeared, Cd is present.

Simple salt test of Sb^{+3}

Antimony is a white metal and its symbol is taken from the Latin name Stibium.

1. $2\ SbCl_3 + 3\ H_2S \longrightarrow Sb_2S_{3\ \text{orange ppt}} + 6\ HCl$

2. $2\ SbCl_3 + 3\ NaOH \longrightarrow Sb(OH)_{3\ \text{white ppt}} + 3\ NaCl$

   $Sb(OH)_3 + NaOH_{excess} \longrightarrow \underset{\text{soluble Sodium antimonite}}{NaSbO_2} + 3\ H_2O$

3. $SbCl_3 + H_2O \longrightarrow \underset{\text{Antimony oxy chloride}}{SbOCl_{\text{white ppt}}} + 2\ HCl$

Simple salt test of Sn^{+2}

1. $SnCl_2 + H_2S \longrightarrow SnS_{\text{brown ppt}} + 2\ HCl$

2. $5\ SnCl_2 + 2\ KMnO_4 \xrightarrow[\text{of } H_2SO_4]{\text{In the presence}} 5\ SnCl_4 + 2\ MnSO_4 + 8\ H_2O$ (colorless)

3. $SnCl_2 + 2\ NaOH \longrightarrow Sn(OH)_{2\ \text{white ppt}} + 2\ NaCl$

   $Sn(OH)_2 + 2\ NaOH_{excess} \longrightarrow \underset{\text{soluble Sodium stannite}}{Na_2SnO_3} + 3\ H_2O$

4. $SnCl_2 + 2\ HgCl_2 \longrightarrow Hg_2Cl_{2\ \text{white ppt}} + SnCl_4$

   $Hg_2Cl_2 + SnCl_{2\ \text{excess}} \longrightarrow 2\ Hg_{\text{black ppt}} + SnCl_4$

Simple salt test of Sn^{+4}

1. $2\ SnCl_4 + 4\ H_2S \longrightarrow Sn_2S_4$ yellow ppt $+ 6\ HCl$

2. $SnCl_4 + 4\ NaOH \longrightarrow Sn(OH)_4$ white ppt $+ 4\ NaCl$

$Sn(OH)_4 + 2\ NaOH_{excess} \longrightarrow Na_2SnO_3 + 3\ H_2O$

soluble Sodium stannite

3. $SnCl_4 + 2\ HgCl_2 \longrightarrow$ No precipitate

Simple salt test of As^{+3}

1. $2Na_3AsO_3 + 3H_2S + 4HCl \longrightarrow As_2S_3$ yellow ppt $+ 6NaCl + 6H_2O$

2. $Na_3AsO_3 + 3\ AgNO_3 \longrightarrow Ag_3AsO_3$ yellow ppt $+ 3NaNO_3$

3. $2Na_3AsO_3 + 3\ CuSO_4 \longrightarrow Cu_3AsO_3$ green yellowish ppt $+ 3NaSO_4$

4. $Na_3AsO_3 + I_2 + H_2O \longrightarrow Na_3AsO_4$ yellow ppt $+ 2HI$

colorless

Simple salt test of As^{+5}

1. $2Na_3AsO_4 + 5H_2S + 6HCl \longrightarrow As_2S_5$ yellow ppt $+ 6NaCl + 8H_2O$

2. $Na_3AsO_4 + 3\ AgNO_3 \longrightarrow Ag_3AsO_4$ brown ppt $+ 3NaNO_3$

3. $2Na_3AsO_4 + 3\ CuSO_4 \longrightarrow Cu_3(AsO_4)_2 + 3NaSO_4$

green blueish ppt

4. $Na_3AsO_4 + NH_4Cl + MgCl_2 \longrightarrow MgNH_4AsO_4 + 3NaCl$

white ppt

Results

Reagent	Sb^{+3}	Sn^{+2}	Sn^{+4}	As^{+3}	As^{+5}
H_2S					
KI					
NaOH					
H_2O					
$SnCl_2$					
$KMnO_4$					
$HgCl_2$					
$AgNO_3$					
$CuSO_4$					
I_2 solution					

Third group

Iron (Fe^{+3}), Chromium (Cr^{+3}), Aluminium (Al^{+3})

Simple salt test of Fe^{+3}

1. $FeCl_3 + 3\ NH_4OH \longrightarrow \underset{\text{redish-brown ppt}}{Fe(OH)_3} + 3\ NH_4Cl$

2. $FeCl_3 + 3\ NaOH \longrightarrow \underset{\text{redish-brown ppt}}{Fe(OH)_3} + 3\ NaCl$

3. $2\ FeCl_3 + 3\ (NH_4)_2S \longrightarrow \underset{\text{black ppt}}{Fe_2S_3} + 6\ NH_4Cl$

4. $FeCl_3 + 3\ K_4[Fe(CN)_6] \longrightarrow \underset{\substack{\text{blue color}\\ \text{Ferric ferro cyanide}}}{Fe_4[Fe(CN)_6]_3} + 12KCl$

5. $FeCl_3 + K_3[Fe(CN)_6] \longrightarrow \underset{\substack{\text{greenish-brown color}\\ \text{Ferric ferric cyanide}}}{Fe[Fe(CN)_6]} + 3KCl$

6. $2\ FeCl_3 + 6\ NH_4SCN \longrightarrow \underset{\text{red-bloody color}}{Fe[Fe(SCN)_6]} + 6\ NH_4Cl$

Simple salt test of Cr^{+3}

1. $CrCl_3 + 3\ NH_4OH \longrightarrow Cr(OH)_3\ \text{Green ppt} + 3\ NH_4Cl$

2. $CrCl_3 + 3\ NaOH \longrightarrow Cr(OH)_3\ \text{Green ppt} + 3\ NaCl$

$$Cr(OH)_3 + 3\ NaOH_{excess} \longrightarrow NaCrO_2 + 2H_2O$$

Green color
of sodium chromite

3. $CrCl_3 + Na_3PO_4 \longrightarrow CrPO_4\ \text{green ppt} + 3\ NaCl$

4. $2\ CrCl_3 + 3\ (NH_4)_2S \longrightarrow Cr_2S_3 + 6\ NH_4Cl$

$$Cr_2S_3 + 6\ H_2O \longrightarrow 2\ Cr(OH)_3\ \text{Green ppt} + 3\ H_2S$$

Chromium sulfide absorb moisture and form green ppt of cromium hydroxide

Simple salt test of Al^{+3}

1. $2Al_2(SO_4)_3 + 3\ Na_2CO_3 \longrightarrow Al_2(CO_3)_3\ \text{white ppt} + 6\ Na_2SO_4$

$$Al_2(CO_3)_3\ \text{white pp} + 3\ H_2O \longrightarrow 2Al(OH)_3\ \text{white ppt} + 3CO_2$$

Aluminium carbonate absorb moisture and form white ppt of aluminium hydroxide

$$Al\ (OH)_3 + Na_2CO_3\ \text{excess} \longrightarrow 2NaAlO_2 + 3H_2O + CO_2$$

Sodium aluminate

2. $2Al_2(SO_4)_3 + 6\ NH_4OH \longrightarrow 2\ Al(OH)_3\ \text{white ppt} + 3(NH_4)_2SO_4$

3. $2Al_2(SO_4)_3 + 6\ NaOH \longrightarrow 2\ Al(OH)_3\ \text{white ppt} + 3Na_2SO_4$

$$Al\ (OH)_3 + NaOH\ \text{excess} \longrightarrow 2NaAlO_2 + 2H_2O$$

Sodium aluminate

4. $Al_2(SO_4)_3 + 3\ (NH_4)_2S \longrightarrow Al_2S_3 + 3(NH_4)_2SO_4$

$$Al_2S_3 + 3\ H_2O \longrightarrow Al(OH)_3\ \text{white ppt} + 3H_2S$$

Aluminium sulfide hydrolyzed and form white ppt of aluminium hydroxide

Results

Reagent	Fe^{+3}	Cr^{+3}	Al^{+3}
NH_4OH			
NaOH			
$(NH_4)_2S$			
H_2S			
$K_4[Fe(CN)_6]$			
$K_3[Fe(CN)_6]$			
NH_4SCN			

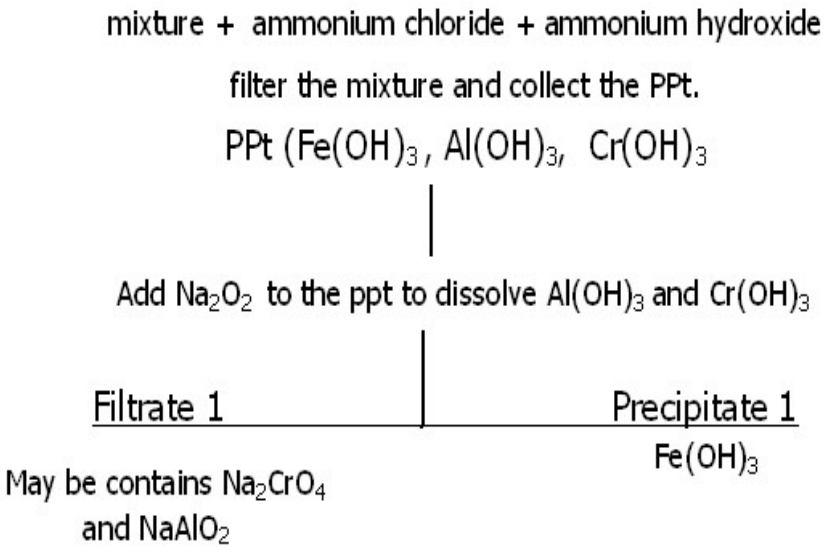
Mixture of the third group

The group will be precipitated according to the solubility product. Hydroxides of the third group have the lowest solubility product comparing with the fourth,….or six group.

We have to use ammonium chloride to control the hydroxide ion.

The group reagent of the third group is ammonium hydroxide (NH_4OH).

The precipitation ion of the third group is hydroxide ion.



1. Dissolve the $Fe(OH)_3$ ppt in diluted HCl, and then add ammonium thiosulfate. If bloody red color appear, the Fe is present.
2. Divide the filtrate into two portions:

   To the first portion, add acetic acid and then lead acetate. If yellow ppt appeared, Cr is present.

   To the second portion, add ammonium chloride and then boil. If white ppt appeared, Al is present.

Fourth group

Zinc (Zn^{+2}), Manganese (Mn^{+2}), Cobalt (Co^{+2}), Nickel (Ni^{+2})

Simple salt test of Zn^{+2}

1. $ZnCl_2 + (NH_4)_2S \longrightarrow ZnS_{\text{white ppt}} + 2NH_4Cl$

   $ZnS + 2\,HCl \longrightarrow ZnCl_2 + H_2S$

Zinc sulfide ppt is formed in case of neutral or alkali solutions but soluble in HCl

2. $ZnCl_2 + H_2S \longrightarrow ZnS_{\text{white ppt}} + 2HCl$

3. $ZnCl_2 + 2\,NaOH \longrightarrow Zn(OH)_{2\,\text{white ppt}} + 2NaCl$

   $Zn(OH)_2 + 2\,NaOH_{excess} \longrightarrow Na_2ZnO_{2\,\text{solible}} + 2H_2O$

4. $ZnCl_2 + NH_4OH \longrightarrow Zn(OH)_{2\,\text{white ppt}} + 2NH_4Cl$

Zinc hydroxide ppt is soluble in excess of ammonium hydroxide

5. $ZnCl_2 + 2\,KCN \longrightarrow Zn(CN)_{2\,\text{white ppt}} + 2KCl$

Zinc cyanide ppt is soluble in excess of KCN

Simple salt test of Mn^{+2}

1. $MnCl_2 + (NH_4)_2S \longrightarrow MnS_{\text{white ppt}} + 2NH_4Cl$

2. $MnCl_2 + H_2S \longrightarrow MnS_{\text{white ppt}} + 2HCl$

3. $MnCl_2 + 2\ NaOH \longrightarrow Mn(OH)_{2\ \text{white ppt}} + 2NaCl$

   $Mn(OH)_{2\ \text{oxidized in air}} + H_2O + O_2 \longrightarrow 4Mn(OH)_{3\ \text{brown}}$

4. $MnCl_2 + 2\ NH_4OH \longrightarrow Mn(OH)_{2\ \text{white ppt}} + 2NH_4Cl$

5. $MnCl_2 + NH_4OH + Na_2PO_4 \longrightarrow Mn(NH_4)PO_{4\ \text{fleshy ppt}} + NaOH$

Simple salt test of Co^{+2}

1. $Co(NO_3)_2 + (NH_4)_2S \longrightarrow CoS_{\text{black ppt}} + 2\ NH_4NO_3$

2. $Co(NO_3)_2 + H_2S \longrightarrow CoS_{\text{black ppt}} + 2HNO_3$

3. $Co(NO_3)_2 + 2\ NaOH \longrightarrow \text{Alkali cobalt salt}_{\text{blue ppt}}$

   $\text{Alkali cobalt salt}_{\text{blue ppt}} + NaOH \xrightarrow{\Delta} Co(OH)_{3\ \text{rose ppt}}$

   The rose ppt change into brown and then black as a result of oxidation

4. $Co(NO_3)_2 + 2\ NH_4OH \longrightarrow \text{Alkali cobalt salt}_{\text{blue ppt}}$

5. $Co(NO_3)_2 + 4\ KSCN \longrightarrow K_2[Co(SCN)_4]_{\text{blue ppt}} + 2KNO_3$

Simple salt test of Ni^{+2}

1. $NiCl_2 + (NH_4)_2S \longrightarrow NiS_{\text{black ppt}} + NH_4Cl$

2. $NiCl_2 + H_2S \longrightarrow NiS_{\text{black ppt}} + 2HCl$

3. $NiCl_2 + 2\ NaOH \longrightarrow Ni(OH)_{2\ \text{green ppt}} + 2NaCl$

4. $NiCl_2 + 2\ NH_4OH \longrightarrow Ni(OH)_{2\ \text{green ppt}} + 2NH_4Cl$

   $Ni(OH)_2 + 6NH_4OH_{\text{excess}} \longrightarrow \underset{\text{blue color}}{[Ni(NH_3)_6](OH)_2} + 6H_2O$

5. $NiCl_2 + K_4[Fe(CN)_6] \longrightarrow Ni_2[Fe(CN)_6]_{\text{yellow ppt}} + 4\ KCl$

Results

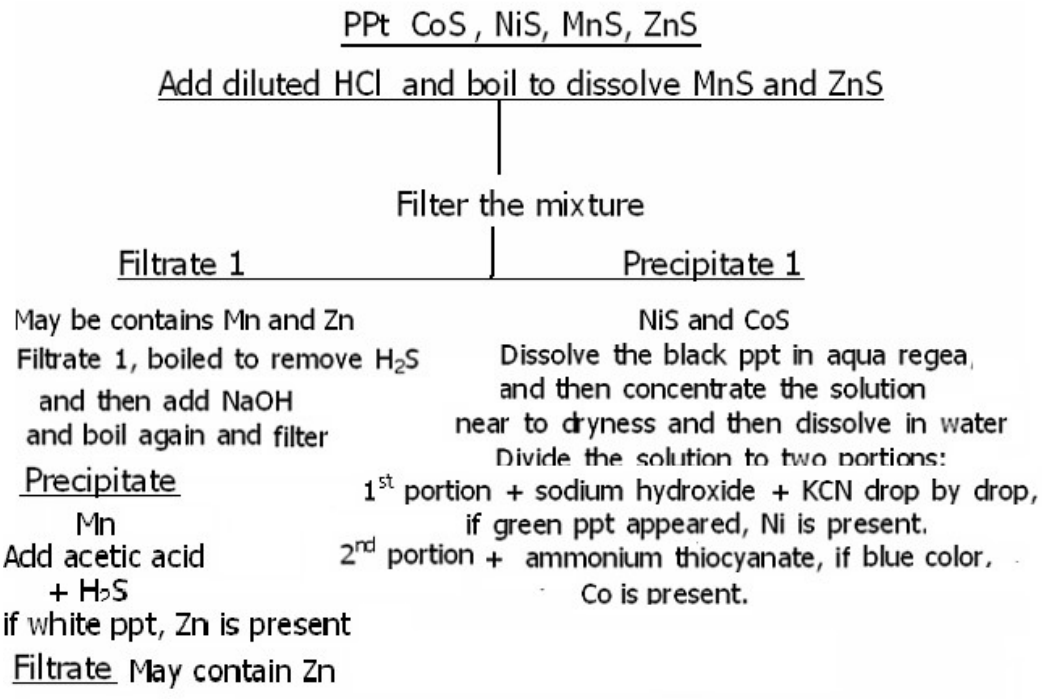
Reagent	Zn^{+2}	Mn^{+2}	Co^{+2}	Ni^{+2}
NH_4OH				
NaOH				
$(NH_4)_2S$				
H_2S				
Na_3PO_4				
$K_3[Fe(CN)_6]$				
NH_4SCN				

Mixture of the fourth group

The group will be precipitated according to the solubility product. Sulfides of the fourth group have the lowest solubility product comparing with the fifth or six group.

The precipitation ion of the fourth group is sulfide ion.

Small amount of mixture in a test tube and then add drops of ammonium chloride, then add ammonium hydroxide and the H_2S. If PPt appeared then add ammonium chloride + ammonium hydroxide + H2S to the all mixture in a beaker and filter the mixture and collect the PPt.



Fifth group

Calcium (Ca^{+2}), Barium (Ba^{+2}), Strontium (Sr^{+2}),

Simple salt test of Ca^{+2}

Calcium is a white silvery metal.

1. $CaCl_2 + (NH_4)_2CO_3 \longrightarrow CaCO_{3\ \text{white ppt}} + 2NH_4Cl$

2. $CaCl_2 + H_2SO_{4\ \text{dil}} \longrightarrow CaSO_{4\ \text{white ppt}} + 2\ HCl$

3. $CaCl_2 + (NH_4)_2C_2O_4 \longrightarrow CaC_2O_{4\ \text{white ppt}} + 2NH_4Cl$

4. $CaCl_2 + K_4[Fe(CN)_6] \longrightarrow CaK_2[Fe(CN)_6]_{\ \text{white ppt}} + 2KCl$

Simple salt test of Sr^{+2}

Strontium is a white silvery metal like calcium.

1. $SrCl_2 + (NH_4)_2CO_3 \longrightarrow SrCO_{3\ \text{white ppt}} + 2NH_4Cl$
2. $SrCl_2 + H_2SO_{4\ \text{dil}} \longrightarrow SrSO_{4\ \text{white ppt}} + 2\ HCl$
3. $SrCl_2 + CaSO_4 \longrightarrow SrSO_{4\ \text{white ppt}} + CaCl_2$
4. $SrCl_2 + K_2CrO_4 \longrightarrow SrCrO_{4\ \text{yellow ppt}} + 2KCl$

strontium chromate is soluble in acetic acid

$SrCrO_4 + 2\ CH_3COOH \longrightarrow (CH_3COO)_2Sr + H_2CrO_4$

5. $SrCl_2 + (NH_4)_2C_2O_4 \longrightarrow SrC_2O_{4\ \text{white ppt}} + 2NH_4Cl$

Simple salt test of Ba^{+2}

1. $BaCl_2 + (NH_4)_2CO_3 \longrightarrow BaCO_{3\ \text{white ppt}} + NH_4Cl$

2. $BaCl_2 + H_2SO_{4\ \text{dil}} \longrightarrow BaSO_{4\ \text{white ppt}} + 2\ HCl$

3. $BaCl_2 + CaSO_4 \longrightarrow BaSO_{4\ \text{white ppt}} + CaCl_2$

4. $BaCl_2 + K_2CrO_4 \longrightarrow BaCrO_{4\ \text{yellow ppt}} + 2KCl$

5. $BaCl_2 + (NH_4)_2C_2O_4 \longrightarrow BaC_2O_{4\ \text{white ppt}} + 2NH_4Cl$

Results

Reagent	Ca^{+2}	Sr^{+2}	Ba^{+2}
$(NH_4)_2CO_3$			
H_2SO_4 dil			
$(NH_4)_2C_2O_4$			
$CaSO_4$			
KSCN			
$K_4[Fe(CN)_6]$			

Mixture of the fifth group

The group will be precipitated according to the solubility product. Carbonates of the fifth group have the lowest solubility product comparing with the sixth group.

The precipitation ion of the fifth group is carbonate ion.

Small amount of mixture in a test tube and then add drops of ammonium hydroxide and then excess amount of ammonium carbonate. Heat the mixture and let it to cool. If PPt appeared filter the mixture.

PPt $BaCO_3$, $CaCO_3$, $SrCO_3$

Add hot acetic acid to dissolve all carbonates

1. Take a small portion and then add Pot. Chromate. If yellow ppt appeared, Ba is present.
2. Take another portion + ammonium hydroxide + ammonium carbonate, if white ppt , Ca or Sr is present. Wash the ppt with hot water and then add hot acetic acid + ammonium hydroxide and divide the mixture to two portions: 1^{st} portion, calcium sulfate or ammonium sulfate, boil and let the mixture for 10 min, if white ppt, Sr is present.
   2^{nd} portion + ammonium oxalate, boil and let the mixture. If white ppt, Ca is present.

Sixth group

Magnesium (Mg^{+2}), Potassium (K^{+}), Sodium (Na^{+}), ammonium (NH_4^{+})

Simple salt test of Mg^{+2}

1. $MgSO_4 + (NH_4)_2CO_3 \xrightarrow{\Delta} MgCO_{3\ \text{white ppt}} + (NH_4)_2\ SO_4$

2. $MgSO_4 + 2\ NH_4OH \longrightarrow Mg(OH)_{2\ \text{white ppt}} + (NH_4)_2\ SO_4$

3. $MgSO_4 + Na_3PO_4 + NH_4Cl \longrightarrow \underset{\text{White ppt}}{Mg(NH_4)PO_4} + Na_2\ SO_4 + NaCl$

Simple salt test of Na^{+}

1. $2\ NaCl + (NH_4)_2C_2O_4 \longrightarrow NaC_2O_{4\ \text{ppt}} + 2\ NH_4Cl$

2. $NaCl + KH_2SbO_4 \longrightarrow NaH_2SbO_{4\ \text{white ppt}} + KCl$

Simple salt test of K^{+}

1. KCl + Tartaric acid $\longrightarrow$ KH tartarate $_{\text{white ppt}}$ + HCl

2. $3\ KCl + Bi(NO_3)_3 + 3\ Na_2S_2O_3 \xrightarrow{\text{ethyl alcohol}} K_3[Bi(S_2O_3)_3]_{\text{yellow ppt}} + 3\ NaNO_3 + 3\ NaCl$

The previous reaction carried out by preparing the mixture of one drop bismuth nitrate + 2-3 drops sodium thiosulfate + 10-15 ml ethanol + potassium chloride. Yellow ppt appeared.

3. $KCl + HClO_4 \longrightarrow KClO_{4\ white\ ppt} + HCl$

Simple salt test of NH_4^+

1. $NH_4Cl + NaOH \longrightarrow NH_4OH + NaCl$

Ammonia gas evolved

$NH_4OH + HCl_{conc} \longrightarrow NH_4Cl + H_2O$

white cloud of ammonium chloride appeared when a glass wet with HCl conc introduced to the ammonia gas evolved.

2. NH_4Cl + sodium tartarate $\longrightarrow$ amm. tartarate $_{white\ ppt}$ + HCl

Results

Reagent	Mg^{+2}	Na^{+}	K^{+}	NH_4^{+}
NH_4OH				
$(NH_4)_2C_2O_4$				
$(NH_4)_2CO_3$				
Sodium tartarate				
NaOH				
$K_3[Fe(CN)_6]$				
KH_2SbO_4				

Identification of acid radicals

The identification of acid radicals could be depends on the volatile products (gases) as a result of treatment of solid sample with acids.

Classification of acid radicals:

1. Radicals which effected with HCl_{dil} or $H_2SO_{4\ dil}$:

Carbonate CO_3^{-2},

Hydrogen carbonate HCO_3^-,

Sulfide S^{-2},

Thiosulfate $S_2O_3^{-2}$,

Sulfite SO_3^{-2},

Nitrite NO_2^-,

Cyanide CN^-,

Cyanates CNO^-

Hypochlorite ClO^-

2. Radicals which effected with $H_2SO_{4\ conc}$:

Chloride Cl^- ,

Bromide Br^- ,

Iodide I^- ,

Nitrate NO_3^-

Thiocyanate SCN^-.

3. Radicals which did not effect with HCl or $H_2SO_{4\ conc}$:

Sulfate SO_4^{-2} ,

Phosphate PO_4^{-3} ,

Borates BO_2^- , $B_4O_7^{-2}$, BO_3^{-3}.

Radicals which effected with HCl_{dil} or $H_2SO_{4\ dil}$

Carbonate CO_3^{-2} and Hydrogen carbonate HCO_3^-

To differentiate between carbonate and hydrogen carbonate, all carbonate tests carried out without heating while hydrogen carbonate happened only with heating.

1. $CO_{3\ \text{solid salt}} + HCl_{dil} \longrightarrow$ Effervescence

$HCO_{3\ \text{solid salt}} + HCl_{dil} \xrightarrow{\Delta}$ Effervescence

$Na_2CO_3 + 2\,HCl \longrightarrow 2NaCl + H_2O + CO_{2\ gas}\uparrow$

$NaHCO_3 + HCl \xrightarrow{\Delta} NaCl + H_2O + CO_{2\ gas}\uparrow$

$CO_{2\ gas} + Ca(OH)_2 \longrightarrow CaCO_{3\ \text{turbidity}} + H_2O$

$CO_{2\ \text{excess}} + CaCO_3 + H_2O \longrightarrow Ca(HCO_3)_{2\ \text{soluble}}$

2. $Na_2CO_3 + MgSO_4 \longrightarrow MgCO_{3\ \text{white ppt}}\downarrow + Na_2SO_4$

$NaHCO_3 + MgSO_4 \xrightarrow{\Delta}$ white ppt $\downarrow$

3. $4Na_2CO_3 + 4HgCl_2 \longrightarrow 3HgO.HgCO_{3\ \text{brown redish ppt}}\downarrow + 8\,NaCl + 3CO_2$

Alkali mercuric carbonate

$NaHCO_3 + HgCl_2 \xrightarrow{\Delta}$ brown redish ppt $\downarrow$

4. $Na_2CO_3 + 2AgNO_3 \longrightarrow Ag_2CO_{3\ \text{white ppt}}\downarrow + NaNO_3$

$NaHCO_3 + AgNO_3 \xrightarrow{\Delta}$ white ppt $\downarrow$

5. $Na_2CO_3 + Ba(NO_3)_2 \longrightarrow BaCO_{3\ \text{white ppt}}\downarrow + NaNO_3$

$NaHCO_3 + Ba(NO_3)_2 \xrightarrow{\Delta}$ white ppt $\downarrow$

Sulfite SO_3^{--}

1. $Na_2SO_{3\ solid\ salt} + 2\,HCl \longrightarrow 2NaCl + H_2O + SO_{2\ gas}\uparrow$

$3SO_{2\ gas} + H_2SO_4 + K_2Cr_2O_7 \longrightarrow K_2SO_4 + Cr_2(SO_4)_{3\ green} + H_2O$

2. $Na_2SO_3 + AgNO_3 \longrightarrow Ag_2SO_{3\ white\ ppt}\downarrow + 2Na\,NO_3$

$2Ag_2SO_3 \xrightarrow{\Delta} Ag_2SO_4 + SO_2 + 2\,Ag_{\ black}\downarrow$

3. $Na_2SO_3 + BaCl_2 \longrightarrow BaSO_{3\ white\ ppt}\downarrow + Na\,Cl$

This white ppt is soluble in diluted HCl.

4. $Na_2SO_3 + Pb(CH_3COO)_2 \longrightarrow 2\,NaCH_3COO + PbSO_{3\ white\ ppt}\downarrow$

This white ppt is soluble in cold diluted HNO_3.

5. $Na_2SO_3 + H_2SO_4 \longrightarrow H_2SO_3 + Na_2SO_4$

$H_2SO_3 + 2\,KMnO_{4\ violet} \longrightarrow K_2SO_4 + MnSO_4 + 2H_2SO_4 + 3H_2O$

The violet color of potassium permanganate is disappeared.

Thiosulfate $S_2O_3^{--}$

1. $Na_2S_2O_{3\ \text{solid salt}} + 2\ HCl \longrightarrow 2NaCl + H_2O + SO_{2\ \text{gas}}\uparrow + S_{\text{yellow}}$

This reaction may need heating.

2. $Na_2S_2O_3 + 2AgNO_{3\ \text{add slowly}} \longrightarrow Ag_2S_2O_{3\ \text{white ppt}}\downarrow + Na\,NO_3$

$Ag_2S_2O_3 + H_2O \longrightarrow H_2SO_4 + Ag_2S_{\text{black}}\downarrow$

3. $Na_2S_2O_3 + Pb(CH_3COO)_2 \longrightarrow 2\,NaCH_3COO + PbS_2O_{3\ \text{white ppt}}\downarrow$

$PbS_2O_{3\ \text{white ppt}} + H_2O \xrightarrow{\text{by boiling}} PbS_{\text{black ppt}}\downarrow + H_2SO_4$

4. $Na_2S_2O_3 + I_2 \longrightarrow 2NaI + Na_2S_4O_6$

The iodine color is disappeared.

Sulfide S^{--}

1. $Na_2S_{\text{solid salt}} + 2\ HCl \longrightarrow 2NaCl + H_2S_{\text{gas}}\uparrow$

H_2S turned wet paper with lead acetate into black

$H_2S + Pb(CH_3COO)_2 \longrightarrow CH_3COOH + PbS_{\text{black ppt}}\downarrow$

2. $Na_2S + 2AgNO_3 \longrightarrow Ag_2S_{\text{black ppt}}\downarrow + 2Na\,NO_3$

$Ag_2S_2O_3 + H_2O \longrightarrow H_2SO_4 + Ag_2S_{\text{black}}\downarrow$

3. $Na_2S + Pb(CH_3COO)_2 \longrightarrow 2\,NaCH_3COO + PbS_{\text{black ppt}}\downarrow$

Nitrite NO_2^-

1. $NaNO_{2\ solid\ salt} + HCl \longrightarrow NaCl + HNO_2$

$3\ HNO_2 \longrightarrow HNO_3 + H_2O + 2\ NO$

$2\ NO + O_{2\ air} \longrightarrow 2NO_2 \uparrow$

2. $2NaNO_2 + 2KI + 2H_2SO_4 \rightarrow Na_2SO_4 + K_2SO_4 + 2NO + I_{2\ brown} + 2H_2O$

3. $NaNO_2 + AgNO_3 \longrightarrow NaNO_3 + AgNO_{2\ white\ ppt} \downarrow$

4. Brown ring experiment

$2NaNO_2 + H_2SO_4 \longrightarrow 2HNO_2 + Na_2SO_4$

$3\ HNO_2 \longrightarrow HNO_3 + H_2O + 2\ NO$

$2\ NO + FeSO_4 \longrightarrow Fe(NO)SO_{4\ brown\ ring}$

Results

Reagent	**CO_3^{-2} HCO_3^-**	**SO_3^-**	**$S_2O_3^{-2}$**	**S^{-2}**	**NO_2^-**
HCl_{dil}	dry salt	dry salt	dry salt	Dry salt	dry salt
$MgSO_4$					
$HgCl_2$					
$AgNO_3$					
NaOH					
$Ba(NO_3)_2$					
$(NH_4)_2C_2O_4$					
Tartaric acid					
$Bi(NO_3)_3$ + $Na_2S_2O_3$					
$HClO_4$					
NaOH					
Nessler solution					
Sodium tartarate					

Radicals which effected with H_2SO_4 conc

Halides, Cl^-, Br-, I^-

1. $2NaCl_{solid} + H_2SO_{4\,conc} \rightarrow Na_2SO_4 + 2HCl\uparrow$

2. $NaBr_{solid} + H_2SO_{4\,conc} \rightarrow NaHSO_4 + HBr\uparrow$

$2HBr + H_2SO_{4\,conc} \rightarrow Br_2\uparrow + 2H_2O + SO_2\uparrow$

3. $2NaI_{solid} + 3H_2SO_{4\,conc} \rightarrow 2NaHSO_4 + 2H_2O + I_2\uparrow + SO_2\uparrow$

4. $NaCl + AgNO_3 \rightarrow Na_2NO_3 + AgCl_{\text{white ppt}}\downarrow$

5. $NaBr + AgNO_3 \rightarrow Na_2NO_3 + AgBr_{\text{yellowish-white ppt}}\downarrow$

6. $NaI + AgNO_3 \rightarrow Na_2NO_3 + AgI_{\text{yellow ppt}}\downarrow$

7. $2NaCl + Pb(CH_3COOH)_2 \rightarrow 2CH_3COONa + PbCl_{2\ \text{white ppt}}\downarrow$

$PbCl_2$ is souble in hot water and precipitated again by cooling

8. $2NaBr + Pb(CH_3COOH)_2 \rightarrow 2CH_3COONa + PbBr_{2\ \text{yellowish-white ppt}}\downarrow$

$PbBr_2$ is souble in hot water and precipitated again by cooling

9. $2NaI + Pb(CH_3COOH)_2 \rightarrow 2CH_3COONa + PbI_{2\ \text{yellow ppt}}\downarrow$

PbI_2 is souble in hot water and precipitated again by cooling

10. $NaCl + Cl_2 \rightarrow$ no reaction

11. $NaBr + Cl_2 \rightarrow 2NaCl + Br_2$

12. $NaI + Cl_2 \rightarrow 2NaCl + I_2$

Nitrate, NO_3^-

1. $NaNO_{3\,solid} + H_2SO_{4\,conc} \rightarrow NaHSO_4 + HNO_3$

$HNO_3 \xrightarrow{\text{dissociated by heating}} 2H_2O + O_2 + NO_2 \uparrow$

2. Brown ring test:

This test may by carried out using two methods:

a) Add 3ml of fresh prepared $FeSO_4$ to 2ml of nitrate solution in a test tube. Add 3-5ml of H_2SO_4 conc slowly with careful on the wall of the test tube till two layer appeared and the brown ring formed between the two layers.

b) Add 4ml of H_2SO_4 conc slowly with careful to 2ml of nitrate solution in a test tube. Mix the mixture and cool the mixture under stream of tap water. Add fresh prepared $FeSO_4$ slowly on the wall of the test tube till two layer appeared and the brown ring formed between the two layers.

$NaNO_3 + H_2SO_4 \rightarrow HNO_3 + NaHSO_4$

$6\,FeSO_4 + 2\,HNO_3 + 3\,H_2SO_4 \rightarrow 3\,Fe(SO_4)_3 + 4\,H_2O + 2\,NO$

$FeSO_4 + NO \rightarrow Fe(NO)SO_{4\,\text{brown ring}}$

Nitrozel

Nitrozel compound (brown ring is unstable and dissociated by heating or shacking and the brown ring disappeared.

Thiocyanate, SCN^-

1. $KSCN_{solid} + 2H_2SO_{4\ conc} \rightarrow$ yellow color and SO_2 gas evolved

2. $KSCN + AgNO_3 \rightarrow KNO_3 + AgSCN_{\text{white ppt}}$

3. $KSCN + CuSO_4 \rightarrow K_2SO_4 + Cu(SCN)_{2\ \text{black ppt}}\downarrow$

4. $2\ KSCN + Hg(NO_3)_2 \rightarrow Hg(SCN)_{2\ \text{white ppt}}\downarrow + 2\ KNO_3$

$Hg(SCN)_{2\ \text{white ppt}}$ very fast soluble in excess of mercuric nitrate.

5. $KSCN + FeCl_3 \rightarrow KCl + [Fe(SCN)]\ Cl_{2\ \text{bloody red color}}$

The red color referred to the ion $[Fe(SCN)]^{+++}$

6. $4\ KSCN + Co(NO_3)_2 \rightarrow 2\ KNO_3 + K_2[Co(SCN)_4]_{\text{blue color}}$

Results

Reagent	**Cl^-**	**Br^-**	**I^-**	**NO_3^-**	**SCN^-**
$H_2SO_{4\ conc}$	dry salt	dry salt	dry salt	Dry salt	dry salt
$AgNO_3$					
$HgCl_2$					
Cl_2					
$Pb(CH_3COO)_2$					
$CuSO_4$					
Brown ring					
$Hg(NO_3)_2$					
$FeCl_3$					
$Co(NO_3)_2$					

Radicals which not effected with acids

Sulfate, SO_4^{-2}

1. $Na_2SO_4 + BaCl_2 \rightarrow BaSO_4\downarrow_{\text{white ppt}} + 2NaCl$

2. $Na_2SO_4 + Pb(CH_3COO)_2 \rightarrow 2\ CH_3COONa + PbSO_4\downarrow_{\text{white ppt}}$

3. $Na_2SO_4 + 2\ AgNO_3 \rightarrow Ag_2SO_4\downarrow_{\text{white ppt}} + 2NaNO_3$

Phosphate, PO_4^{-3}

1. $Na_2HPO_4 + BaCl_2 \rightarrow BaHPO_4\downarrow_{\text{white ppt}} + 2NaCl$

2. $Na_2HPO_4 + FeCl_3 + NH_4OH \rightarrow FePO_4\downarrow + NH_4Cl + 2\ NaCl + H_2O$

yellowish white ppt

3. $2\ Na_2HPO_4 + 3\ AgNO_3 \rightarrow Ag_3PO_4\downarrow_{\text{yellow ppt}} + 3NaNO_3 + NaH_2PO_4$

4. $Na_2HPO_4 +$ ammonium molybdate solutior. $+ HNO_3 \xrightarrow{\Delta}$ ammonium phospho molybdate$\downarrow$

yellow ppt

Borate, $B_4O_7^{-2}$

1. $Na_2B_4O_7 + BaCl_2 \longrightarrow 2\,Ba(BO_2)_{2\ white\ ppt}\downarrow + 2NaCl$

$Ba(BO_2)_{2\ white\ ppt}$ is soluble in excess of barium chloride

2. $Na_2B_4O_7 + 2\,AgNO_3 + H_2O \longrightarrow 2\,AgBO_{2\ white\ ppt}\downarrow + 2\,NaNO_3 + 2\,H_3BO_3$

Results

Reagent	**SO_4^{-2}**	**PO_4^{-3}**	**$B_4O_7^{-2}$**
$AgNO_3$			
$FeCl_3$			
$BaCl_2$			
$Pb(CH_3COO)_2$			
Ammonium molybdate			

Safety Rules in Chemical Laboratories

General rules inside the chemical laboratory:

- You have to come in time without delay.
- White coat used to protect your clothes from any deterioration.
- You have to study and understand the experiment very well.
- Do not touch, smell, or taste any material or chemical
- Use the pipette filler to take any portion of liquid chemical, do not suck with your mouth.
- Do not inhale the vapors of chemicals or solvents.
- Do not waste in consuming chemicals.
- Do not eat or drink inside the lab.
- Put the flammable solvents away from fire.
- Be careful during heating or boiling any chemical in a test tube
- All chemical reactions and processes which release vapors or gases should be carried out inside the hood.
- To dilute the concentrated acids, it should be added to the water gradually and carefully with stirring. Do not add water to the acid.
- The distillation processes for flammable solvents should be done in closed system using electrical bath water. Do not use direct flame.

The sources of danger inside the laboratory:

1. Toxic chemicals.
2. Flammable chemicals.
3. Explosive chemicals.
4. Glassware.

First aid inside the chemical laboratory:

Acid burning:

- Wash the burnt area with a gentle stream of water.
- Wash the burnt area with 5% of sodium hydrogen carbonate solution.
- Wash with water again.

Alkali burning:

- Wash the burnt area with a gentle stream of water.
- Wash the burnt area with 5% of ammonium chloride solution.
- Wash with water again.

Phenol or bromine irritating:

- Wash the irritated area with organic solvent , e.g., alcohol,ether, benzene.

Flame burning:

- Wash the burnt area with 5% potassium permenganate.
- In case of slightly burning, wash with alcohol and put layer of glycerin.

Eye hurtful:

- If alkali material reach the eye, wash eye with a lot of water and then wash with 2% boric acid solution.
- If acid material reach the eye, wash eye with saturated solution of borax or 5% of sodium hydrogen carbonate solution.
- You have to go to hospital if any accident happened to eye.

Precautions to avoid harmful during the handling of glassware:

- Take care during inserting thermometer or glass tube into hole of rubber plug. You can wet the tip of tube with glycerol or water.
- Use silicone grease to connect two or more pieces of glass.

Chemical label:

- Read the label of chemicals carefully.
- Take only the amount you need in a beaker or test tube.
- Do not deteriorate the chemicals by returning the remained amount to the chemical bottle .
- Cap the bottle directly after taking the amount you need.
- There are important special symbols you have to be aware during the handling of different chemicals:

Corrosion material

Dangerous vapour

Flammable

Explosive material

Toxic material

Radioactive material

Glassware in Laboratories

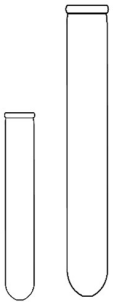
Glassware used in lab are resist to chemicals, easy to follow up the reaction and easy to clean.

Glassware divided into two categories:

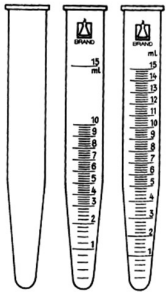
First, Glassware used in heating: This type tolerant for heating even with direct flam but can not used in measuring.

Examples:

1. Test tubes

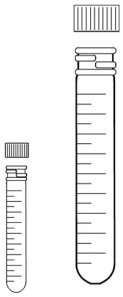


Regular test tubes

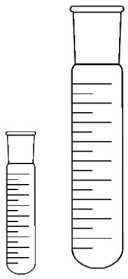


Conical test tubes

Burning tubes



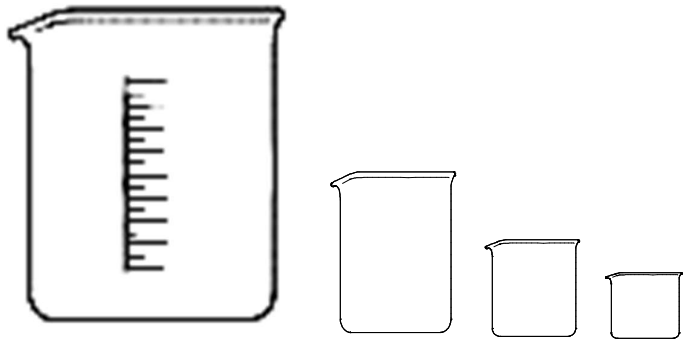
Capped test tubes



Graduated test tubes

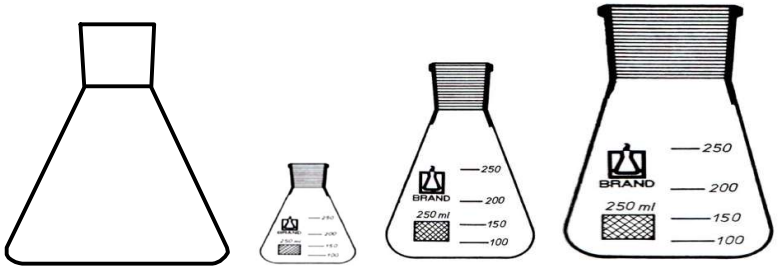
2. Beakers

They are used in solution preparation, liquid heating and precipitation processes.



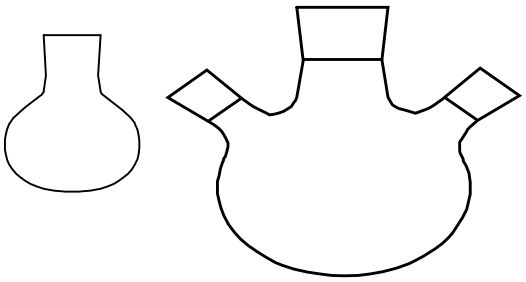
3. Conical Flasks

Conical flasks are used in titration, heating, and filtration processes.



4. Round bottom flasks

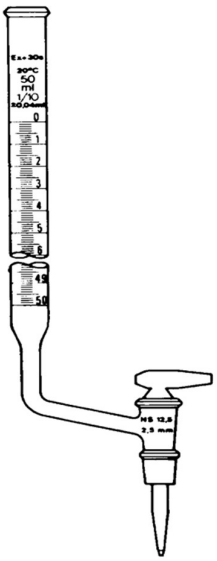
They are used in preparation of chemical reactions, extraction and distillation processes.



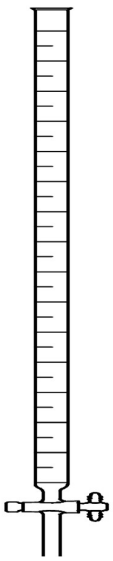
Second, Glassware used in Measuring: This type is used only in measuring process but does not tolerant for heating processes.

Examples:

1. Burette: It is used in volumetric titration.

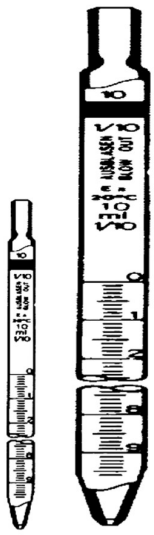


Graduated burette

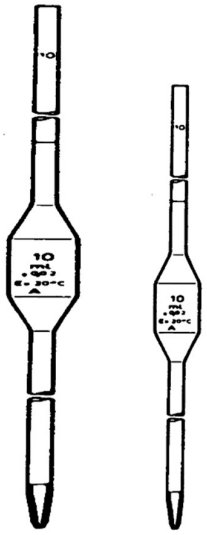


Volumetric burette

2. Pipette: It is used to take a certain volume of liquids.

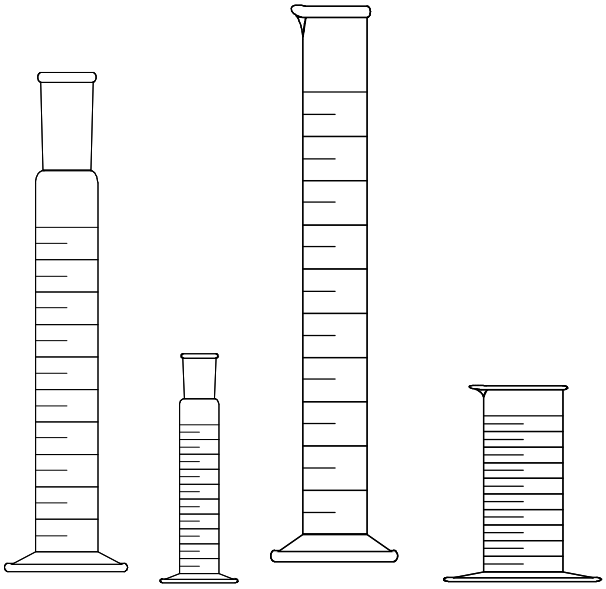


Graduated pipette

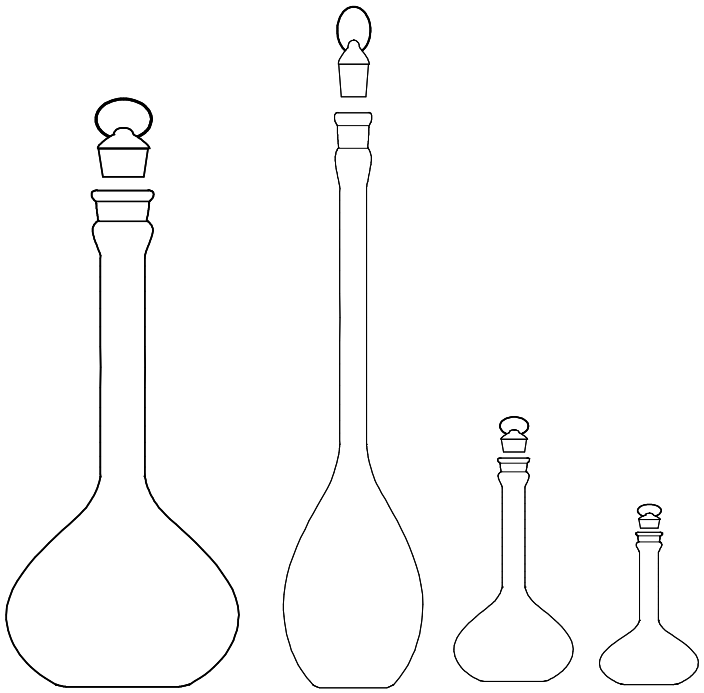


Volumetric pipette

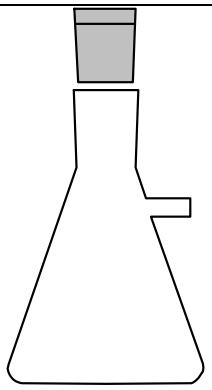
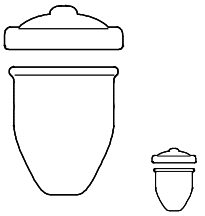
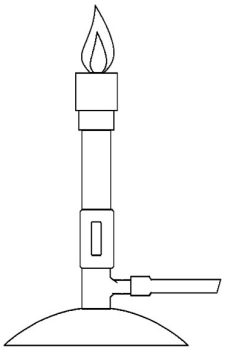
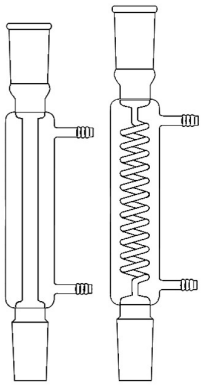
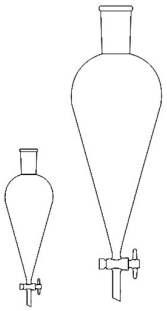
3. Graduated cylinder: It is used to take a roughly volume of liquids.

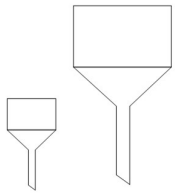


4. Volumetric flask: It is used for preparation of concentrations of solutions

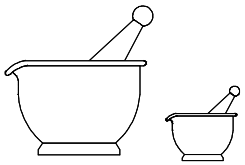


Shapes of some tools and glasses used in chemistry lab

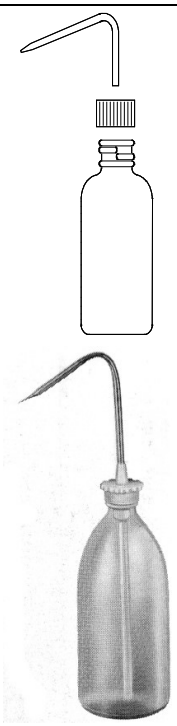
 Filtering flask	 Indicator bottle	 crucible
 Bunsen burner	 condenser	 Separatory funnel



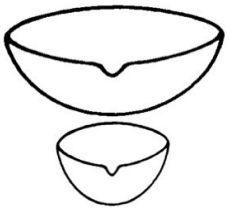
funnel Büchner



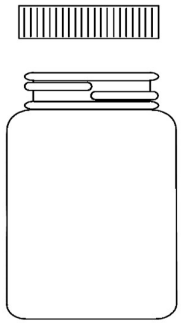
Mortar



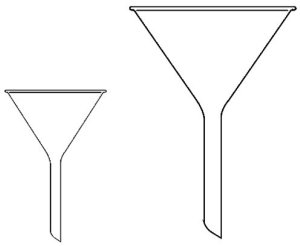
Wash bottle



Evaporating Dish

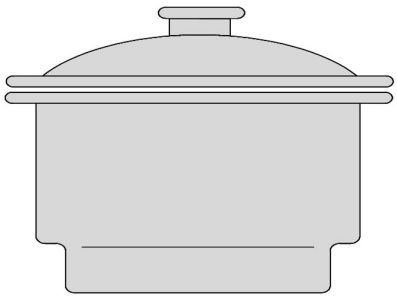


Reagent bottle



funnel

Clamp stand, support



Desicator

Balance

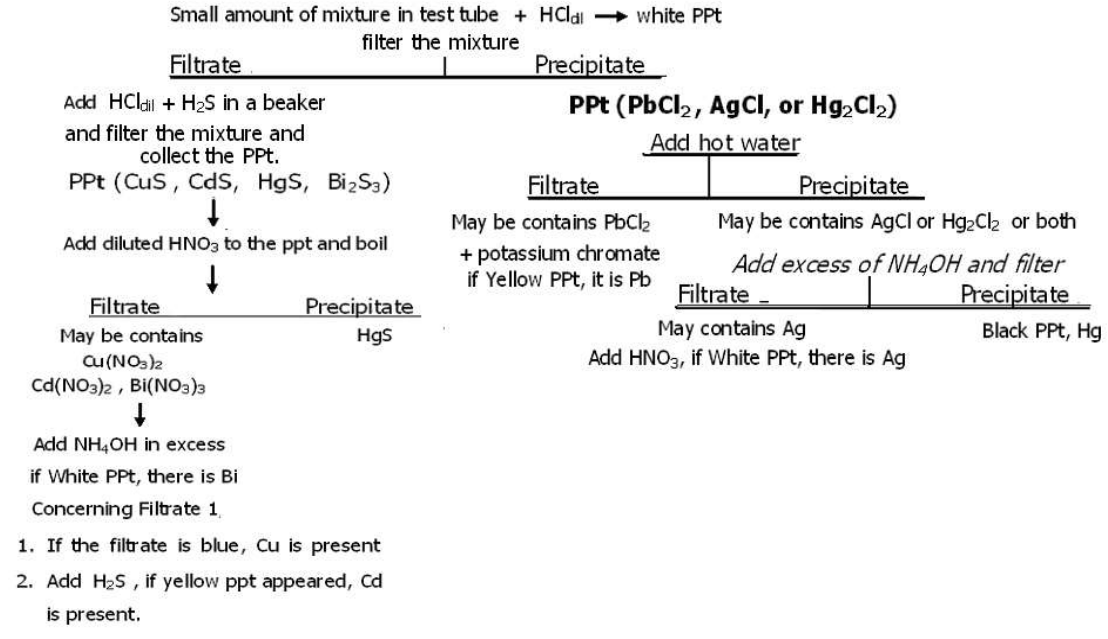
Greek prefix	Number	Greek prefix	Number
Mono-	1	Hexa-	6
Di-	2	Hepta-	7
Tri-	3	Octa-	8
Tetra-	4	Nona-	9
Penta-	5	Deca-	10

Name	Formula	Name	Formula
Acetate	$CH_3CO_2^-$	Cyanide	CN^-
Ammonium	NH_4^+	Hydroxide	HO^-
Carbonate	CO_3^{2-}	Nitrate	NO_3^-
Chlorate	ClO_3^-	Permanganate	MnO_4^-

Chromate	CrO_4^{2-}	Phosphate	PO_4^{3-}
Dichromate	$Cr_2O_7^{2-}$	Sulfate	SO_4^{2-}

Iron	Ferrous, Fe^{2+}	Ferric, Fe^{3+}
Copper	Cuprous, Cu^{+}	Cupric, Cu^{2+}
Tin	Stannous, Sn^{2+}	Stannic, $Sn4^{+}$
Lead	Plumbous, Pb^{2+}	Plumbic, Pb^{4+}
Mercury	Mercurous, Hg^{+}	Mercuric, Hg^{2+}

***-ic* acid**	**Name**	***-ous* acid**	**Name**
HCl	Hydrochloric acid		
HBr	Hydrobromic acid		
HCN	Hydrocyanic acid		
H_2CO_3	Carbonic acid		
H_2SO_4	Sulfuric acid	H_2SO_3	Sulfurous acid
HNO_3	Nitric acid	HNO_2	Nitrous acid
H_3PO_4	Phosphoric acid	H_3PO_3	Phosphorous acid
$HClO_3$	Chloric acid	$HClO_2$	Chlorous acid
$HClO_4$	Perc`hloric acid	HClO	Hypochlorous acid


Small amount of mixture in test tube + HCl_{dl} → white PPt
filter the mixture
Filtrate
Precipitate
Add HCl_{dil} + H_2S in a beaker and filter the mixture and collect the PPt.
PPt (CuS , CdS, HgS, Bi_2S_3)
Add diluted HNO_3 to the ppt and boil
Filtrate
Precipitate
May be contains $Cu(NO_3)_2$ $Cd(NO_3)_2$, $Bi(NO_3)_3$
HgS
Add NH_4OH in excess
if White PPt, there is Bi
Concerning Filtrate 1
1. If the filtrate is blue, Cu is present
2. Add H_2S , if yellow ppt appeared, Cd is present.
PPt ($PbCl_2$, AgCl, or Hg_2Cl_2)
Add hot water
Filtrate
Precipitate
May be contains $PbCl_2$ + potassium chromate if Yellow PPt, it is Pb
May be contains AgCl or Hg_2Cl_2 or both
Add excess of NH_4OH and filter
Filtrate
Precipitate
May contains Ag
Add HNO_3, if White PPt, there is Ag
Black PPt, Hg

mixture + ammonium chloride + ammonium hydroxide

PPt ($Fe(OH)_3$, $Al(OH)_3$, $Cr(OH)_3$

Filtrate 1	Precipitate 1
May be contains Na_2CrO_4 and $NaAlO_2$	$Fe(OH)_3$
add ammonium chloride + ammonium hydroxide + H2S	Dissolve the $Fe(OH)_3$ ppt in diluted HCl, and then add ammonium thiosulfate. If bloody red color appear, the Fe is present.
PPt CoS, NiS, MnS, ZnS	

Add diluted HCl and boil to dissolve MnS and ZnS

Filter the mixture

Filtrate	Precipitate
May be contains Mn and Zn	NiS and CoS
Filtrate 1, boiled to remove H_2S and then add NaOH and boil again and filter	Dissolve the black ppt in aqua regea, and then concentrate the solution near to dryness and then dissolve in water
Precipitate: Mn	Divide the solution to two portions:
Add acetic acid + H_2S	1st portion + sodium hydroxide + KCN drop by drop, if green ppt appeared, Ni is present.
if white ppt, Zn is present	2nd portion + ammonium thiocyanate, if blue color, Co is present.
Filtrate: May contain Zn	

add drops of ammonium hydroxide and then excess amount of ammonium carbonate.

PPt $BaCO_3$, $CaCO_3$, $SrCO_3$

Add hot acetic acid to dissolve all carbonates

1. Take a small portion and then add Pot. Chromate. If yellow ppt appeared, Ba is present.
2. Take another portion + ammonium hydroxide + ammonium carbonate, if white ppt, Ca or Sr is present. Wash the ppt with hot water and then add hot acetic acid + ammonium hydroxide and divide the mixture to two portions: 1st portion, calcium sulfate or ammonium sulfate, boil and let the mixture for 10 min, if white ppt, Sr is present.
   2nd portion + ammonium oxalate, boil and let the mixture. If white ppt, Ca is present.