Republic of Iraq
Ministry of Higher Education and ScientificResearch
Mustansiriyah University
Collage of Science-Department of Chemistry

Practice Qualitative Chemical Analysis

First Grade

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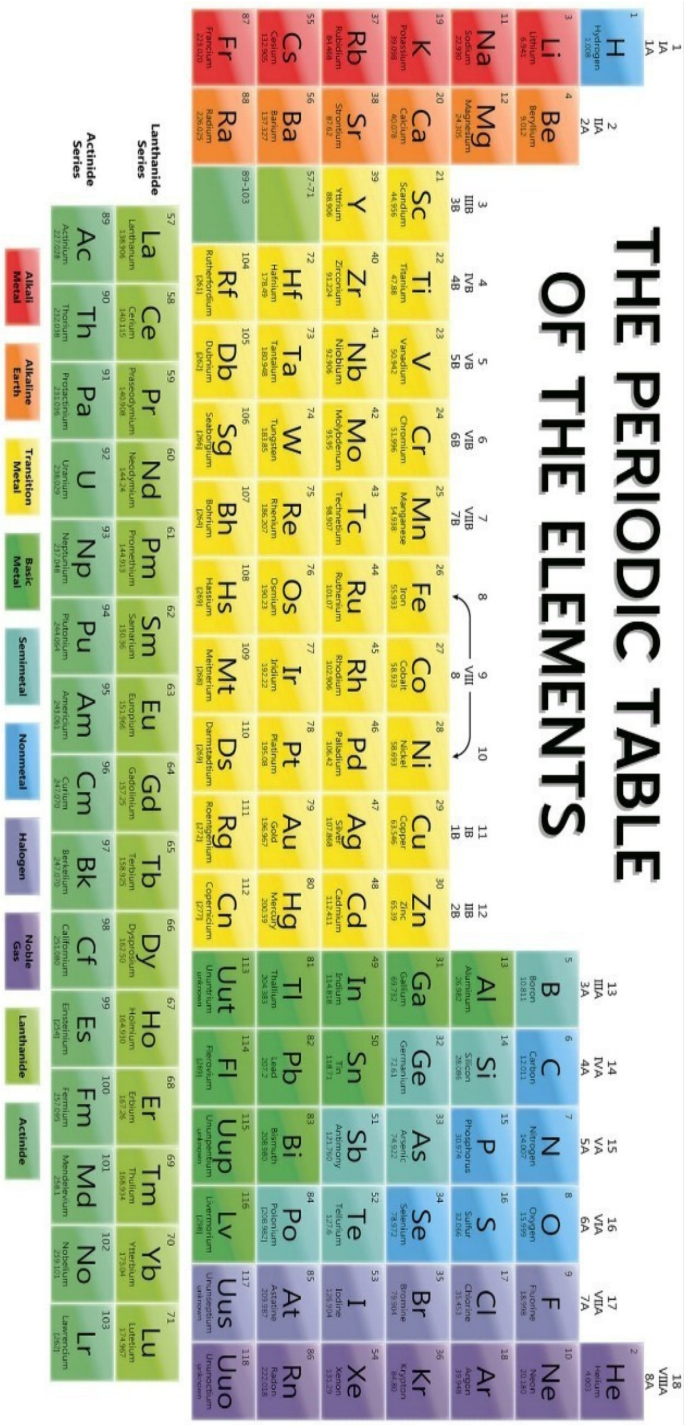

THE PERIODIC TABLE OF THE ELEMENTS
Lanthanide Series
Actinide Series
Alkali Metal
Alkaline Earth
Transition Metal
Basic Metal
Semimetal
Nonmetal
Halogen
Noble Gas
Lanthanide
Actinide

Experiment no (2)

Separation and analysis of group 2 cations (arsenic- copper group)

Copper group **(Cu^{+2}, Cd^{+2}, Hg^{+2}, Pb^{+2}, Bi^{+3}) IIA**

Arsenic group **(Sb^{+3}, As^{+3}, Sn^{+4}) IIB**

Ions of this group share the possibility of precipitation and separation on the form of sulphides from a solution of 0.3 M for HCl. The H_2S gas is used as a precipitation agent or Aqueous Solution ofthioacetamide (CH_3CSNH_3).It will be a source of H_2S gas release. Thioacetamide dissolves easily with water and then aqueous analysis especially when the temperature is higher than the room temperature and according to the equation

$$CH_3CSNH_2 + H_2O \longrightarrow CH_3CONH_2 + H_2S \uparrow$$

There is another way to generate H_2S gas continuously using HCl concentrated in a Kipp device where the reaction can be easily controlled starting or stopping the reaction as soon as the gas faucet is opened and closed, this can be explainedaccording to the equation below:

$$FeS + 2HCl \longrightarrow FeCl_2 + H_2S$$

Precipitation using a thioacetamide solution is much easier and better than using a very toxic H_2S gas. The use of H_2S gas in the precipitation requires the entry of gas in the solution inside the gas cabinet and pay attention to the gas in the solution in order not to lose part of the precipitate solution. Using the thioacetamide solution requires adding enough drops of this solution to (the group or ion) solution to be precipitate and then heating the solution produced inside the test tube in a water bath until the complete precipitate phase. It is important to note here, that not only are the group 2 sulfides are not dissolved in water, but a number of heavy element ions that are precipitated as sulfides, which fall within the five groups in the qualitativeanalysis. The second group of positive ions (copper-arsenic group) includes eight ions that are precipitate and separated as Sulfides form. Of the HCl acid solution, H_3O^+ concentration in it ranges from 0.2-0.3 MThe precipitation factor used is either H_2S or solution thioacetamide (TA). After the precipitation of this group usingthe above conditions, it will be easy to isolated from

the rest of the subsequent groups (Third, fourth and fifth). Here is a large difference in the solubility product of the large differencebetween the values of water-constrained constants in the table below. Two groups of sulphides are precipitate in the acidic solution (group 2 ions sulfides) and the other is precipitate from a basic solution.

The following table shows the insoluble sulphides of some positive ions of the second and third groups with the values of the solubility product constants.

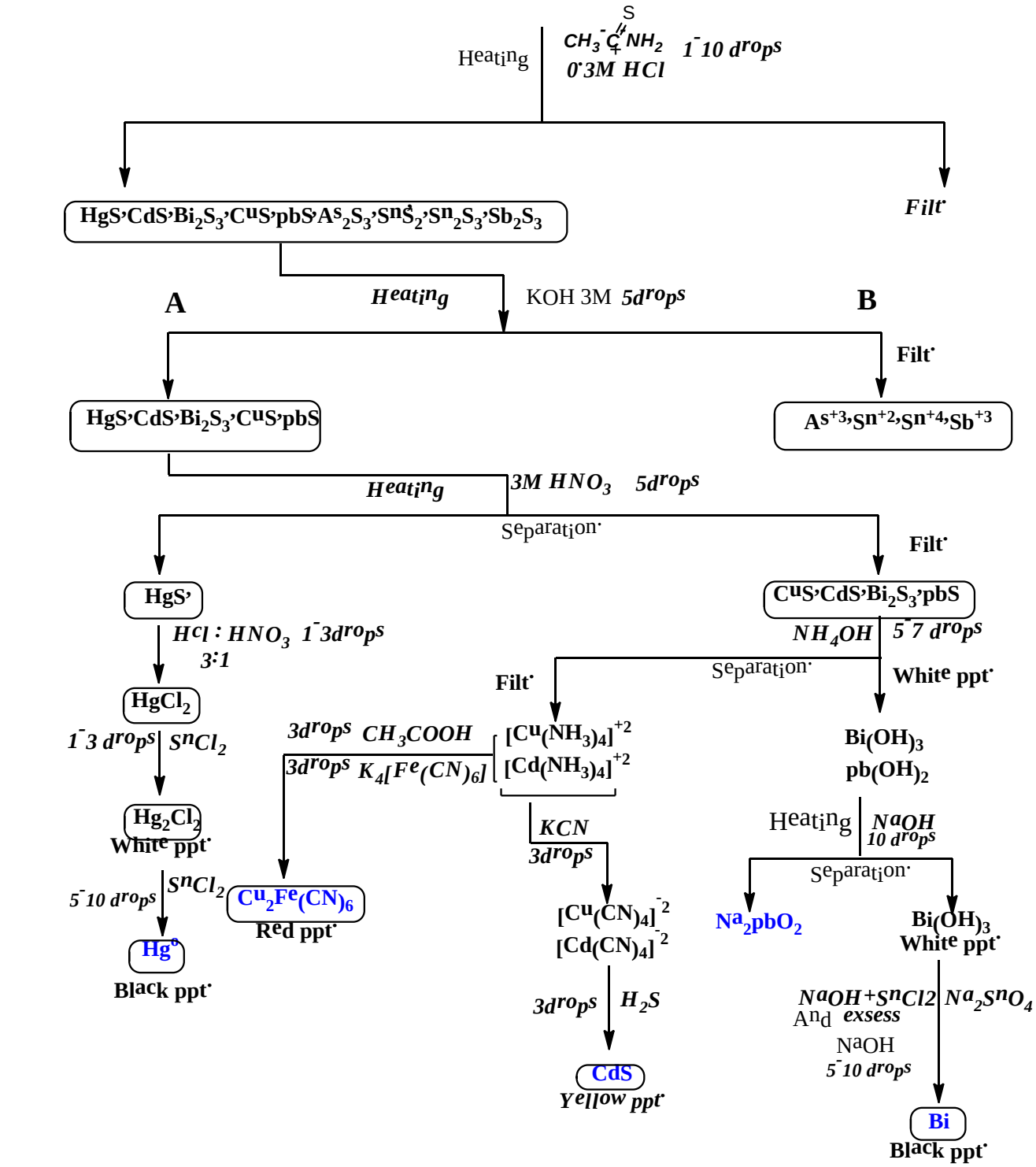
Ks.p	The sulphide	Ks.p	The sulphide
1.4×10^{-15}	MnS	3.6×10^{-29}	CdS
3.7×10^{-19}	FeS	$\mathbf{8.5} \times 10^{-45}$	CuS
7.0×10^{-23}	CoS	3.0×10^{-55}	HgS
1.2×10^{-23}	ZnS	1.6×10^{-72}	Bi2S3
1.4×10^{-24}	NiS	1.9×10^{-85}	Sb2S3

Procedure:

1. We have a mix of group 2 cations (arsenic- copper group). Add 3 drops thioacetamide (TA) solution and 3 drops HNO_3.to a mixture of group 2 cations(arsenic- copper group).
2. Preheat in water bath for about approximatelya quarter of an houruntil a black Precipitate appears to convert to a brown color by heating.
3. Separate the filtrate(I)from the Precipitate(I).
4. The resultedprecipitate (I) consists of(HgS).Therefore, add (Aqua regia)and heated in a water bath with stirring.The precipitate dissolves and we obtain the Hg^{+2}ion was obtained. After that, add 3 dropsof $SnCl_2$ solution (reduced agent) until it turns intoa black precipitate from Hg^{o} and stop adding it. At the end we have detected the presence of mercury ion (Hg^{+2}) in the mix.
5. The filtrate (I) contains these ions(Cu^{+2}, Cd^{+2}, Pb^{+2}, Bi^{+3}). Add (3-5) drops of ammonia solution. A white precipitate appears consistinga consist of Bi $(OH)_3$ and Pb $(OH)_2$.

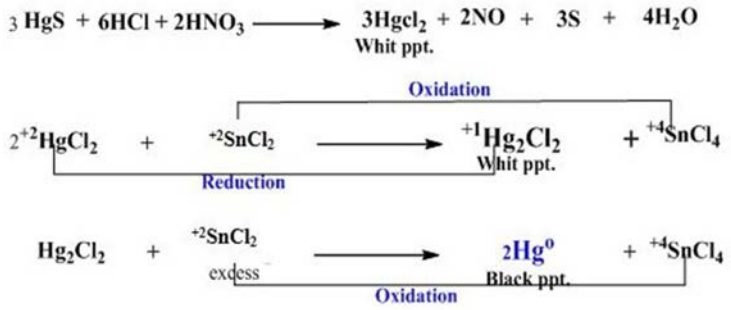
6. Separate the filtrate (II) from the Precipitate (II).
7. Add 2 drops of NaOH solution with stirring to the Precipitate (II) and place it in the centrifuge to obtain a precipitate (III) and filtrate (III).
8. The white precipitate (III) is $Bi(OH)_3$ added to its sodium stannite reagent and turns into a black color indicating the presence Bi^{+3} ion.
9. **Preparation of sodium stannite reagent**from the reaction of 3 drops of NaOH solution with an increase of $SnCl_2$ solution until a white precipitator is formed to indicate the formation of sodium stannite reagent.
10. The filtrate (III) Consists of the Na_2PbO_4, add K_2CrO_4 potassium chromate solution to turn into $PbCrO_4$ yellow color indicating the presence Pb^{+2}ion.
11. The filtrate (II) contains $Cu[NH_3]_4]^{+2}$, $[Cd(NH_3)_4]^{+2}$ divided it into two parts (AII) and (BII).
12. Add 3 drops from KCN solution to The filtrate)AII (to turn into solution contain of $[Cu(CN)_4]^{+2}$ and $[Cd(CN)_4]^{+2}$ add to it 3 drops of thioacetamide (TA) solution and then heat in water bath until a yellow precipitate is formed from CdS compound, indicating the presence Cd^{+2} ion
13. Add 3 drops from acetic acid CH_3COOH solution and 3 drops from Potassium ferrocyanide$K_4[Fe(CN)_6]$ solution to The filtrate (BII) to turn into red solution due to the formation of $Cu_2[Fe(CN)_6]$ complex indicating the presence Cu^{+2} ion.

SEPARATION AND ANALYSIS OF THE SECOND GROUPII

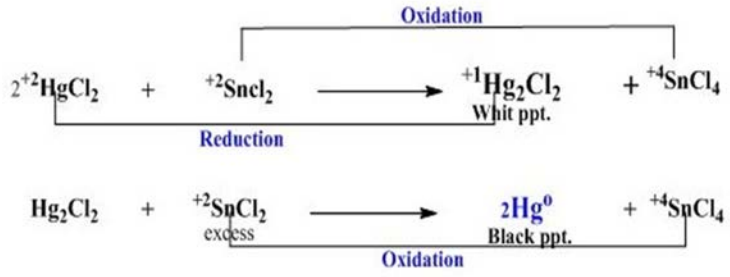


EQUATIONS OF SEPARATION AND ANALYSIS OF THE SECOND GROUP II

Detection equations for Hg^{+2} ion



Equations of oxidation and reduction of Hg^{+2} ions



Interaction equations $Cu^{+2}Cd^{+2}$ with NH_4OH

Detection equations for Cu^{+2} ion

$$[Cu(NH_3)_4]^{+2} + K_4[Fe(CN)_6] \xrightarrow{CH_3COOH} Cu_4[Fe(CN)_6$$

Red ppt.

Interaction equations of complex with KCN

$$\begin{matrix}[Cu(NH_3)_4]^{+2} \\ [Cd(NH_3)_4]^{+2}\end{matrix} + KCN \longrightarrow \begin{matrix}[Cu(CN)_4]^{-2} \\ [Cd(CN)_4]^{-2}\end{matrix}$$

Detection equations for Cd^{+2} ion

$$[Cd(CN)_4]^{-2} + H_2S \longrightarrow CdS$$

Yellow ppt.

Composition equations of *Bismuth* hydroxide and *Lead* hydroxide

$$Bi^{+3} + \underbrace{NH3 + H_2O}_{NH_4OH} \longrightarrow Bi(OH)_3 + NH_4^+$$

White ppt.

$$pb^{+2} + \underbrace{NH3 + H_2O}_{NH_2OH} \longrightarrow pb(OH)_2 + NH_4^+$$

White ppt.

Preparation equations Na_2SnO_2

$$SnCl_2 + 2NaOH \longrightarrow \underset{\text{White ppt.}}{Sn(OH)_2} + 2NaCl$$

$$Sn(OH)_2 + 2NaOH \longrightarrow \underline{Na_2SnO_2} + 2H_2O$$

Not: The disappearance of the white ppt. Indication of being Na_2SnO_2

Equation detected ion Bi^{+3}

$$\underset{\text{or } 2Bi(NO_3)_3}{3Bi^{+3}} + 2NaOH + Na_2SnO_2 \longrightarrow \underset{\text{Black ppt.}}{Bi^0\downarrow} + 3Na_2SnO_3 + 6NaNO_3 + 3H_2O$$