Republic of Iraq
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Practice Qualitative Chemical Analysis

First Grade - First Term

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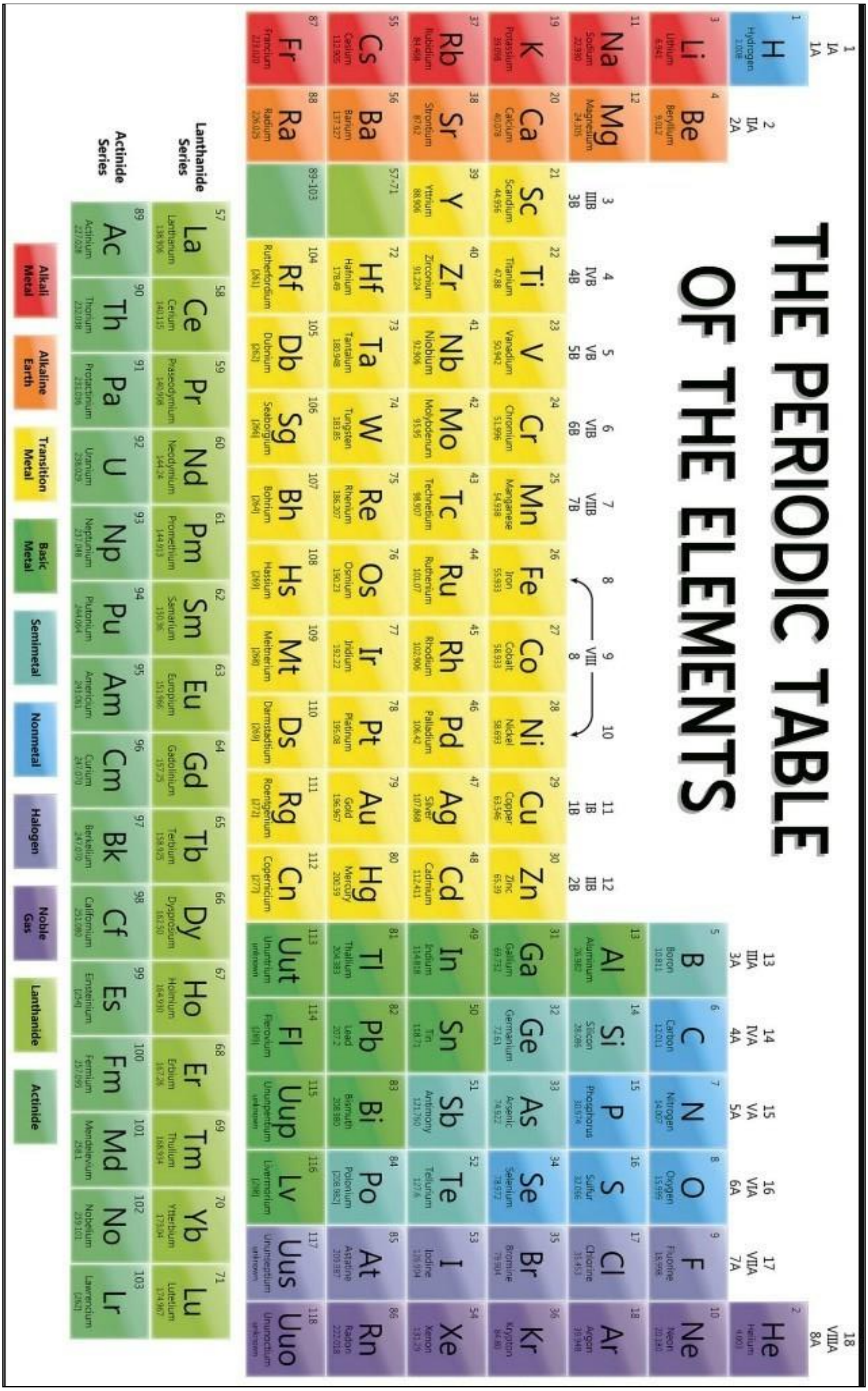

THE PERIODIC TABLE OF THE ELEMENTS
Lanthanide Series
Actinide Series
Alkali Metal
Alkaline Earth
Transition Metal
Basic Metal
Semimetal
Nonmetal
Halogen
Noble Gas
Lanthanide
Actinide

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**

Theoretical bases

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 of thioacetamide (CH_3CSNH_3) It will be a source of H_2S gas release. Thioacetamide it 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, and according to the equation

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

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 precipitate as sulfides, which fall within the five groups in the qualitative analysis . 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 M The precipitation factor used is

either H_2S or solution thioacetamide (TA) After the precipitation of this group in the above conditions will be easily isolated from the rest of the subsequent groups (Third, fourth and fifth). Here is a large difference in the solubility product of the large difference between 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.
2. Preheat in water bath About a quarter of an hour a black Precipitate (I) appears convert to a brown color by heating.
3. separate the filtrate (I) from the Precipitate (I).
4. Precipitate (I) Consists of (HgS) Add (Aqua regia) And heated in a water bath with stirring The precipitate dissolves and we obtain the Hg^{+2} ion. After that, add drops of SnCl2 solution (reduced agent) until it turns into a black precipitate from Hg^{o} and stop adding it Thus we have detected the presence of mercury ion (Hg^{+2}) in the mix.
5. The filtrate (I) contains of this ions (Cu^{+2}, Cd^{+2}, Pb^{+2}, Bi^{+3}) add (3-5) drops of ammonia solution. A white precipitate appears a 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.

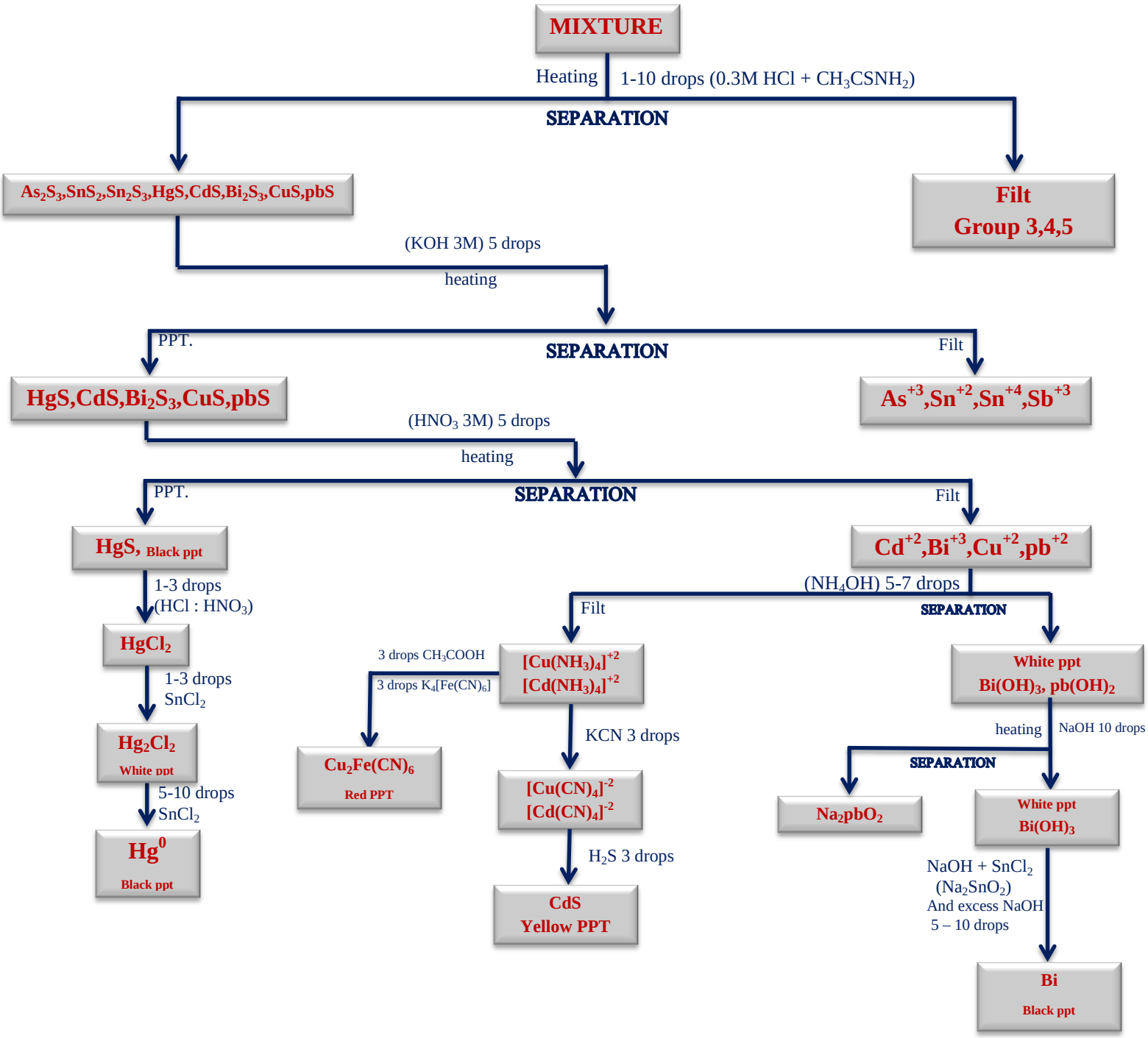
10. The filtrate (III) Consists of Na_2PbO_4 add K_2CrO_4 potassium chromate solution to turn into $PbCrO_4$ yellow color indicating the presence Pb^{+2} ion.

11. The filtrate (II) contains Cu [NH3] 4] +2 , [Cd (NH3) 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 formation of $Cu_2[Fe(CN)_6]$ complex indicating the presence Cu^{+2} ion

SEPARATION AND ANALYSIS OF THE SECOND GROUP II



الكشف العام لأيونات الطائفة الثانية (مجموعة النحاس)

$Hg^{+2} + H_2S \rightarrow HgS + 2H^{+1}$

$Cu^{+2} + H_2S \rightarrow CuS + 2H^{+1}$

$Cd^{+2} + H_2S \rightarrow CdS + 2H^{+1}$

$Pb^{+2} + H_2S \rightarrow PbS + 2H^{+1}$

$2Bi^{+3} + 3H_2S \rightarrow Bi_2S_3 + 6H^{+1}$

الكشف العام لأيونات الطائفة الثانية (مجموعة الزرنيخ)

$2As^{+3} + 3H_2S \rightarrow As_2S_3 + 6H^{+1}$

$Sb^{+3} + 3H_2S \rightarrow Sb_2S_3 + 6H^{+1}$

$Sn^{+2} + H_2S \rightarrow SnS + 2H^{+1}$

$Sn^{+4} + 2H_2S \rightarrow SnS_2 + 4H^{+1}$

الكشف التأكيدي لأيون الزئبقيك

$3HgS + 6HCl + 2HNO_3 \rightarrow HgCl_2 + 2NO + 3S + 4H_2O$

$HgCl_2 + SnCl_2 \rightarrow Hg_2Cl_2 + SnCl_4$

$Hg_2Cl_2 + SnCl_{2\ (excees)} \rightarrow Hg_{(black\ ppt)} + SnCl_4$

الكشف التأكيدي لأيون النحاس

$$Cu^{+2} + NH_4OH \rightarrow [Cu(NH_3)_4]^{+2}$$

$$[Cu(NH_3)_4]^{+2} + K_4[Fe(CN)_6] \xrightarrow{CH3COOH} Cu_2\,Fe(CN)_6\ (\text{red ppt})$$

الكشف التأكيدي لأيون الكادميوم

$$Cd^{+2} + NH_4OH \rightarrow [Cd(NH_3)_4]^{+2}$$

$$[Cd(NH_3)_4]^{+2} + KCN \rightarrow [Cd(CN)_4]^{-2}$$

$$[Cd(CN)_4]^{-2} + H_2S \rightarrow CdS_{(\text{yellow ppt})}$$

تحضير قصديريت الصوديوم

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

$$Sn(OH)_2 + 2NaOH \rightarrow Na_2SnO_2 + 2\,H_2O$$

الكشف التأكيدي لأيون البزموث

$$2Bi^{+3} + 3Na_2SnO_2 + 6NaOH \rightarrow 2Bi_{(\text{black ppt})} + 3\,Na_2SnO_3 + 3H_2O$$