

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



Practice Qualitative Chemical Analysis

First Grade

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THE PERIODIC TABLE OF THE ELEMENTS

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|--------------------------------------|---------------------------------------|---|---|---------------------------------------|---|--|---|--|--|---|---|--|--|--|--|--|--|------------------------------------|------------------------------------|-------------------------------------|------------------------------------|---------------------------------------|-------------------------------------|
| 1 IA H Hydrogen 1.008 | 2 IIA He Helium 4.003 | | | | | | | | | | | | | | | | | 13 IIIA B Boron 10.811 | 14 IVA C Carbon 12.011 | 15 VA N Nitrogen 14.007 | 16 VIA O Oxygen 15.999 | 17 VIIA F Fluorine 18.998 | 18 VIIIA Ne Neon 20.180 |
| 3 IIIA Li Lithium 6.941 | 4 IIA Be Beryllium 9.012 | 5 IIIA B Boron 10.811 | 6 IVA C Carbon 12.011 | 7 VA N Nitrogen 14.007 | 8 VIA O Oxygen 15.999 | 9 VIIA F Fluorine 18.998 | 10 VIIIA Ne Neon 20.180 | 11 IB Cu Copper 63.546 | 12 IIB Zn Zinc 65.38 | 13 IIIA Al Aluminum 26.982 | 14 IVA Si Silicon 28.086 | 15 VA P Phosphorus 30.974 | 16 VIA S Sulfur 32.06 | 17 VIIA Cl Chlorine 35.45 | 18 VIIIA Ar Argon 39.948 | | | | | | | | |
| 19 IA K Potassium 39.098 | 20 IIA Ca Calcium 40.078 | 21 IIIB Sc Scandium 44.956 | 22 IVB Ti Titanium 47.88 | 23 VB V Vanadium 50.942 | 24 VIB Cr Chromium 51.996 | 25 VIIB Mn Manganese 54.938 | 26 VIII Fe Iron 55.845 | 27 VIII Co Cobalt 58.933 | 28 VIII Ni Nickel 58.693 | 29 VIII Cu Copper 63.546 | 30 VIII Zn Zinc 65.38 | 31 IIIA Ga Gallium 69.723 | 32 IVA Ge Germanium 72.61 | 33 VA As Arsenic 74.922 | 34 VIA Se Selenium 78.972 | 35 VIIA Br Bromine 79.904 | 36 VIIIA Kr Krypton 83.80 | | | | | | |
| 37 IA Rb Rubidium 85.468 | 38 IIA Sr Strontium 87.62 | 39 IIIB Y Yttrium 88.906 | 40 IVB Zr Zirconium 91.224 | 41 VB Nb Niobium 92.906 | 42 VIB Mo Molybdenum 95.94 | 43 VIIB Tc Technetium 98.906 | 44 VIII Ru Ruthenium 101.07 | 45 VIII Rh Rhodium 102.905 | 46 VIII Pd Palladium 106.367 | 47 VIII Ag Silver 107.868 | 48 VIII Cd Cadmium 112.411 | 49 IIIA In Indium 114.818 | 50 IVA Sn Tin 118.71 | 51 VA Sb Antimony 121.757 | 52 VIA Te Tellurium 127.6 | 53 VIIA I Iodine 126.905 | 54 VIIIA Xe Xenon 131.29 | | | | | | |
| 55 IA Cs Cesium 132.905 | 56 IIA Ba Barium 137.327 | 57 IIIB La Lanthanum 138.905 | 58 IVB Hf Hafnium 178.49 | 59 VB Ta Tantalum 180.948 | 60 VIB W Tungsten 183.84 | 61 VIIB Re Rhenium 186.207 | 62 VIII Os Osmium 190.23 | 63 VIII Ir Iridium 192.222 | 64 VIII Pt Platinum 195.084 | 65 VIII Au Gold 196.967 | 66 VIII Hg Mercury 200.59 | 67 IIIA Tl Thallium 204.38 | 68 IVA Pb Lead 207.2 | 69 VA Bi Bismuth 208.98 | 70 VIA Po Polonium 209 | 71 VIIA At Astatine 210 | 72 VIIIA Rn Radon 222 | | | | | | |
| 87 IA Fr Francium 223 | 88 IIA Ra Radium 226 | 89-103 Lanthanide Series La Lanthanum 138.905 Ce Cerium 140.12 Pr Praseodymium 140.908 Nd Neodymium 144.24 Pm Promethium 144.913 Sm Samarium 150.36 Eu Europium 151.964 Gd Gadolinium 157.25 Tb Terbium 158.925 Dy Dysprosium 162.50 Ho Holmium 164.930 Er Erbium 167.257 Tm Thulium 168.930 Yb Ytterbium 173.054 Lu Lutetium 174.967 | 104 IVB Rf Rutherfordium 101.07 | 105 VB Db Dubnium 102.906 | 106 VIB Sg Seaborgium 106.905 | 107 VIIB Bh Bohrium 107.904 | 108 VIII Hs Hassium 108.906 | 109 VIII Mt Meitnerium 108.906 | 110 VIII Ds Darmstadtium 110.906 | 111 VIII Rg Roentgenium 111.906 | 112 VIII Cn Copernicium 112.906 | 113 IIIA Uut Ununtrium 113.906 | 114 IVA Fl Flerovium 114.906 | 115 VA Uup Ununpentium 115.906 | 116 VIA Lv Livermorium 116.906 | 117 VIIA Uus Ununseptium 117.906 | 118 VIIIA Uuo Ununoctium 118.906 | | | | | | |

| | | | | | | | | | | | |
|--------------------------|------------------------|---------------------|-----------------------|-------------------------|--------------------|-------------------|-----------------|----------------|------------------|-------------------|-----------------|
| Lanthanide Series | Actinide Series | Alkali Metal | Alkaline Earth | Transition Metal | Basic Metal | Semi-metal | Nonmetal | Halogen | Noble Gas | Lanthanide | Actinide |
|--------------------------|------------------------|---------------------|-----------------------|-------------------------|--------------------|-------------------|-----------------|----------------|------------------|-------------------|-----------------|

Experiment no (2)***Separation and analysis of group 2 cations (arsenic- copper group)***

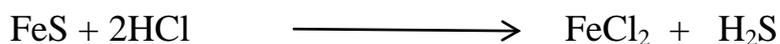
Copper group (Cu^{+2} , Cd^{+2} , Hg^{+2} , Pb^{+2} , Bi^{+3}) IIA

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 of thioacetamide (CH_3CSNH_2). 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



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 explained according to the equation below:



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 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 using the 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 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.

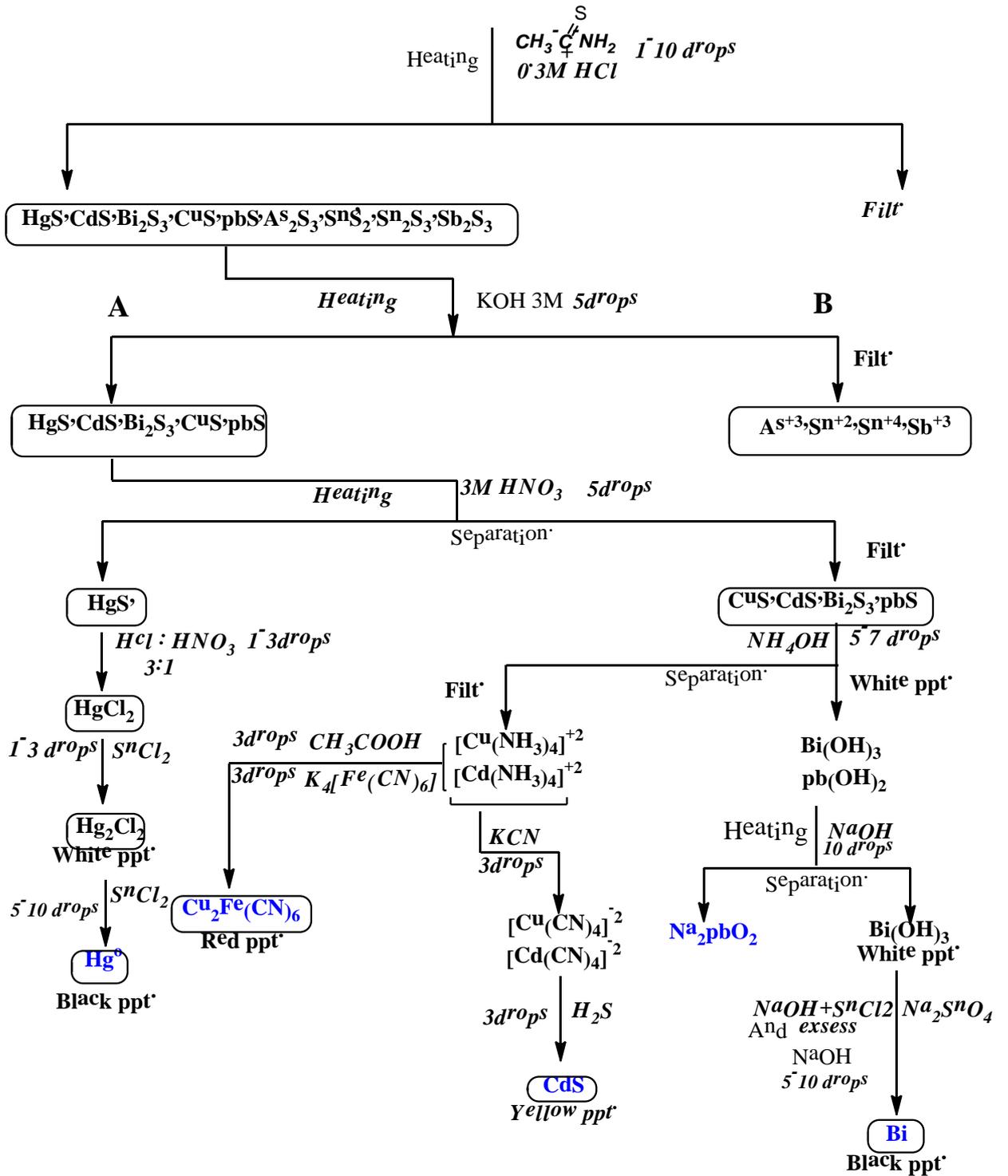
| Ks.p | The sulphide | Ks.p | The sulphide |
|-----------------------|--------------|-----------------------|--------------------------------|
| 1.4×10^{-15} | MnS | 3.6×10^{-29} | CdS |
| 3.7×10^{-19} | FeS | 8.5×10^{-45} | CuS |
| 7.0×10^{-23} | CoS | 3.0×10^{-55} | HgS |
| 1.2×10^{-23} | ZnS | 1.6×10^{-72} | Bi ₂ S ₃ |
| 1.4×10^{-24} | NiS | 1.9×10^{-85} | Sb ₂ S ₃ |

Procedure:

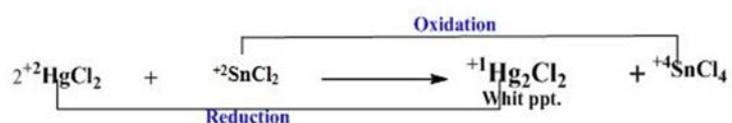
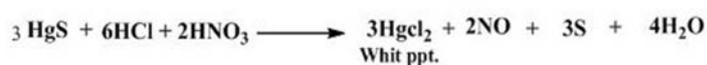
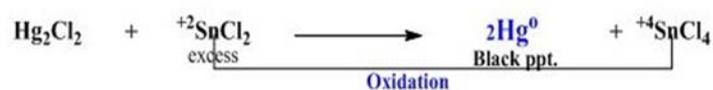
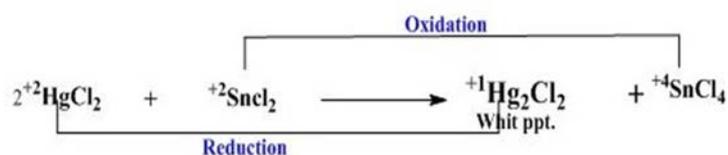
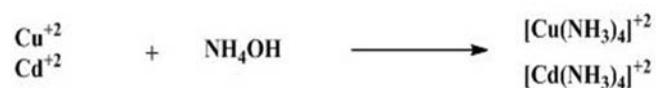
1. We have a mix of group 2 cations (arsenic- copper group). Add 3 drops thioacetamide (TA) solution and 3 drops HNO₃ to a mixture of group 2 cations(arsenic- copper group).
2. Preheat in water bath for about approximately a quarter of an hour until a black Precipitate appears to convert to a brown color by heating.
3. Separate the filtrate(I) from the Precipitate(I).
4. The resulted precipitate (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 drops of SnCl₂ solution (reduced agent) until it turns into a black precipitate from Hg⁰ 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 consisting of Bi (OH)₃ and Pb (OH)₂.

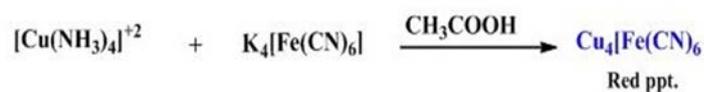
6. Separate the filtrate (II) from the Precipitate (II).
7. Add 2 drops of NaOH solution with stirring to the Precipitate (II) and place it in the centrifuge to obtain a precipitate (III) and filtrate (III).
8. The white precipitate (III) is $\text{Bi}(\text{OH})_3$ added to its sodium stannite reagent 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 precipitate 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 $\text{Cu}[\text{NH}_3]_4^{+2}$, $[\text{Cd}(\text{NH}_3)_4]^{+2}$ 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 $[\text{Cu}(\text{CN})_4]^{+2}$ and $[\text{Cd}(\text{CN})_4]^{+2}$ 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 $\text{K}_4[\text{Fe}(\text{CN})_6]$ solution to The filtrate (BII) to turn into red solution due to the formation of $\text{Cu}_2[\text{Fe}(\text{CN})_6]$ complex indicating the presence Cu^{+2} ion.

SEPARATION AND ANALYSIS OF THE SECOND GROUP II

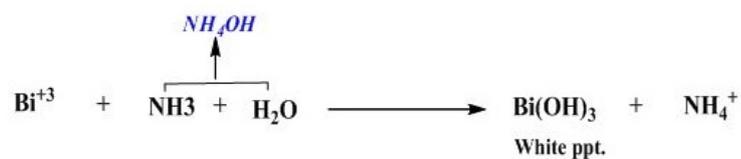


EQUATIONS OF SEPARATION AND ANALYSIS OF THE SECOND GROUP II

Detection equations for Hg^{+2} ionEquations of oxidation and reduction of Hg^{+2} ionsInteraction equations $\text{Cu}^{+2}\text{Cd}^{+2}$ with NH_4OH 

Detection equations for Cu^{+2} ion

Interaction equations of complex with KCN

Detection equations for Cd^{+2} ionComposition equations of *Bismuth* hydroxide and *Lead* hydroxide

Preparation equations Na_2SnO_2 

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

Equation detected ion Bi^{+3} 