Second lab
Analytical Chemistry

Saba Abd Ul-munemHabeeb
Ms.c Applied Chemistry

2017

Second lab
Analytical Chemistry

The science of the methods for studying the composition of material. It consists of two basic divisions:

- ✓ Qualitative analysis .
- ✓ Quantitative analysis.

Qualitative analysis .

- Qualitative analysis consists of methods for establishing the qualitative chemical composition of a substance—that is, the identification of atoms, ions, and molecules that enter into the composition of the substance being analyzed.

- The most important characteristics of all methods of qualitative analysis are specificity and sensitivity. Specificity characterizes the ability to detect the presence of an unknown element in the presence of other elements—for example, iron in the presence of nickel, manganese, chromium, vanadium, or silicon.

Quantitative analysis

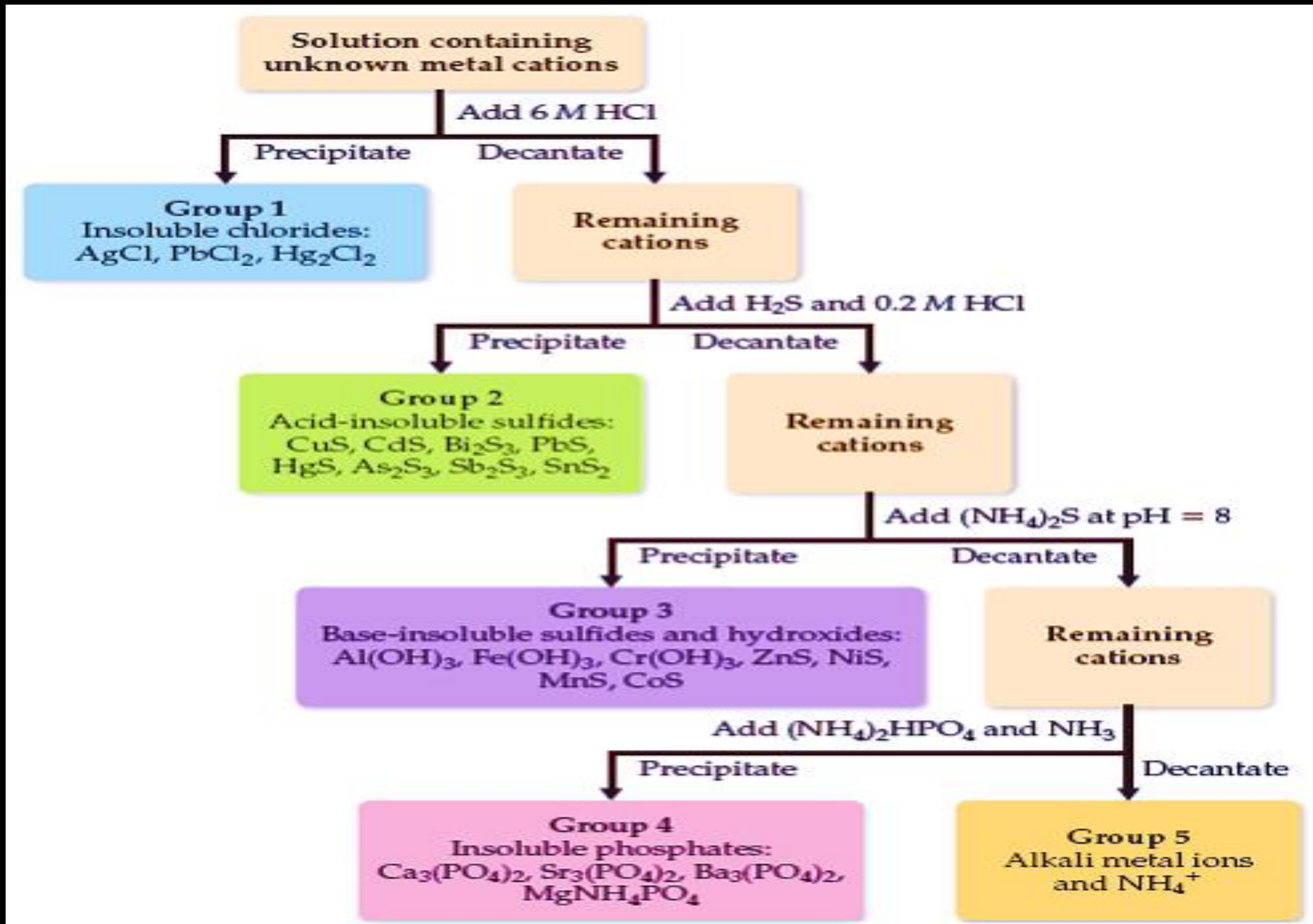
- The quantitative amounts of the chemical elements or of certain compounds in the analyzed substance. Along with specificity and sensitivity, the most important characteristic of every method of quantitative analysis is accuracy.
- The accuracy of an analysis is expressed in a percentage of error, which in most cases must not exceed 1–2 percent.
- Sensitivity in quantitative analysis is expressed in percentages

Qualitative Analysis of Group 1 Cations

(ANALYSIS OF THE SILVER GROUP CATIONS)

Ag^{+}, Pb^{2+} and Hg_2^{2+}

Cations are typically divided into **Groups**, where each group shares a common reagent that can be used for selective precipitation


Solution containing unknown metal cations
Add 6 M HCl
Precipitate
Decantate
Group 1
Insoluble chlorides:
$AgCl$, $PbCl_2$, Hg_2Cl_2
Remaining cations
Add H_2S and 0.2 M HCl
Precipitate
Decantate
Group 2
Acid-insoluble sulfides:
CuS, CdS, Bi_2S_3, PbS, HgS, As_2S_3, Sb_2S_3, SnS_2
Remaining cations
Add $(NH_4)_2S$ at pH = 8
Precipitate
Decantate
Group 3
Base-insoluble sulfides and hydroxides:
$Al(OH)_3$, $Fe(OH)_3$, $Cr(OH)_3$, ZnS, NiS, MnS, CoS
Remaining cations
Add $(NH_4)_2HPO_4$ and NH_3
Precipitate
Decantate
Group 4
Insoluble phosphates:
$Ca_3(PO_4)_2$, $Sr_3(PO_4)_2$, $Ba_3(PO_4)_2$, $MgNH_4PO_4$
Group 5
Alkali metal ions and NH_4^+

- Since these ions all form insoluble chlorides, their separation from all other ions may be accomplished by the addition of 6 M HCl (aq) resulting in the precipitation of AgCl (s), PbCl2 (s), and Hg_2Cl_2 (s):

- ❑ $Ag^+ (aq) + Cl^- (aq) \rightarrow AgCl (s)$
- ❑ $Hg_2^{2+} (aq) + 2\ Cl^- (aq) \rightarrow Hg_2Cl_2 (s)$
- ❑ $Pb_2^+ (aq) + 2\ Cl- (aq) \rightarrow PbCl_2 (s)$

Separation and Confirmation of Group 1 Cations

- Lead(II) chloride can be separated from the other two chlorides based on its increased solubility at higher temperatures. This means that lead(II) chloride will dissolve in hot water, leaving the mercury(I) chloride and the silver chloride in solid form:
- $PbCl_2 (s) \rightarrow Pb^{2+} (aq) + 2\ Cl^{-} (aq)$

- The presence of Pb^{2+} in the aqueous solution can then be confirmed by the formation of a yellow precipitate of PbCrO4 upon the addition of aqueous K2CrO4:
- $Pb^{2+} (aq) + CrO_4^{2-} (aq) \rightarrow PbCrO_4 (s)$

- Next, Hg_2^{2+} and Ag^+ cations can be separated by adding 6 M NH_3 (aq) to the solid mixture of the two chlorides. Silver chloride will dissolve since it forms a soluble complex ion with ammonia:
- $AgCl$ (s) + 2 NH_3 (aq) → $Ag(NH_3)^{2+}$ (aq) + Cl^- (aq)
- However, mercury(I) chloride reacts with the ammonia yielding what appears to be a gray solid which is actually a mixture of black Hg (l) and white $HgNH_2Cl$ (s). The presence of this gray solid is confirmation of the presence of Hg_2^{2+}.
- Hg_2Cl_2 (s) + 2 NH_3 (aq) → Hg (l) + HgNH2Cl (s) + NH4+ (aq) + Cl- (aq)

- The presence of **Ag⁺** can be confirmed by the appearance of a white precipitate upon adding 6 M HNO_3 (aq) to the solution. The nitric acid reacts with the ammonia and thus destroys the complex ion containing the silver cation. Once in solution again the silver cation precipitates with the chloride as indicated by the following reactions:

✓ $Ag(NH_3)^{2+}$ (aq) + 2 H^+ (aq) → Ag^+ (aq) + 2 NH^{4+} (aq)

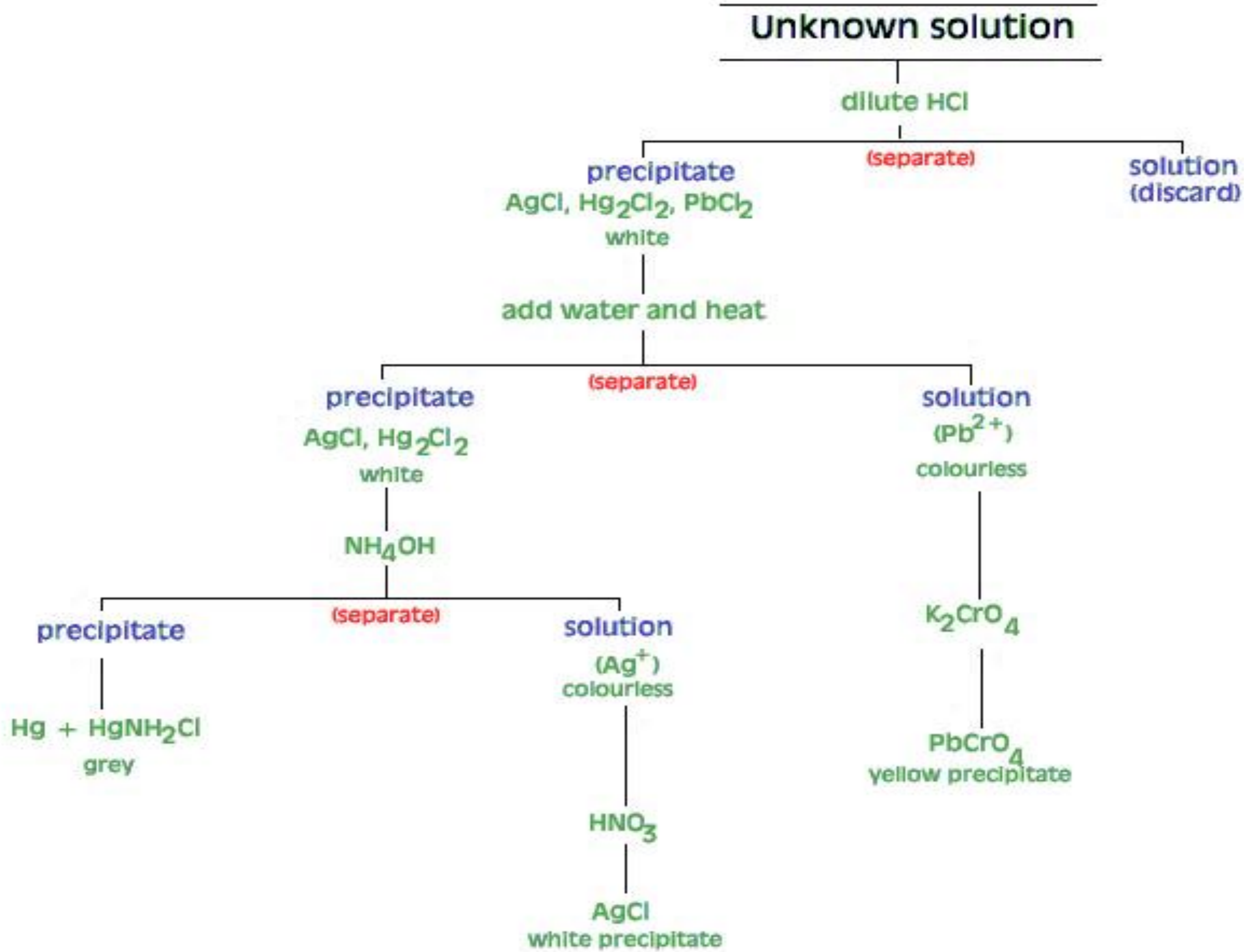
✓ Ag^+ (aq) + Cl^- (aq) → AgCl (s)

(a) Precipitation of group 1 cations by adding HCl(aq): $AgCl(s) + Hg_2Cl_2(s) + PbCl_2(s)$

(b) Confirmation test for silver by addition of $Na_2CrO_4(aq)$: $PbCrO_4(s)$

(c) Confirmation test for mercury by adding $NH_3(aq)$: $Hg(l) + Hg(NH_2)Cl(s)$

(d) Confirmation test for lead by by adding HCl(aq): AgCl(s)



Unknown solution
dilute HCl
(separate)
precipitate
AgCl, Hg2Cl2, PbCl2
white
solution
(discard)
add water and heat
(separate)
precipitate
AgCl, Hg2Cl2
white
solution
(Pb2+)
colourless
NH4OH
(separate)
precipitate
Hg + HgNH2Cl
grey
solution
(Ag+)
colourless
HNO3
AgCl
white precipitate
K2CrO4
PbCrO4
yellow precipitate

Lab repots

Title of Experiment:

Student Name: ***Group:*** ***Date:***

		Reagents				
Solution	Metal					
Lead nitrate (II) **$Pb(NO_3)_2$**						
Sliver nitrate (I) **AgNO3**						
Mercury nitrate (II) **$(Hg(NO_3)_2$**						

- ***Lab safety*** for {(Lead nitrate (II)$Pb(NO_3)_2$) ,(Sliver nitrate (I) $AgNO_3$) ,(Mercury nitrate(II) $(Hg(NO_3)_2$)} , please describe the lab safety for all salts by samples ?
- ***Discussion***