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



# Practice Qualitative Chemical Analysis First Grade

**Edited by** 

Alaa Abullwahid Jasim Ali Amer Waheb Ruba Fahmi Abass Neda Ibrahim Mahdi

Assistant lecturer

Supervised by

Dr.Khitam Jaber Nabhan

**Dr.Amer Saleh Mahdi** 

Lecturer

Lecturer

	A Li
Lambanic scriedsS7 L L L MaxS6 C C C MaxS6 P P P MaxS60 P P MaxS7 P P MaxS60 P P P MaxS60 P P P MaxS60 P P P MaxS60 P P P MaxS60 P <th>THE P THE P TH</th>	THE P THE P TH
Edd Gal Straight     65 Tuby Tission     66 Dup Tuby Tission     66 Dup TubyTission     66 Dup TubyTission     66 Dup TubyTission <t< th=""><th>TS IIII</th></t<>	TS IIII
Y FI 68 69   Yornow Example 100   Y Yornow 100   F ES Fm N   Non- Energian Nergian Nergian   Non- Energian Nergian Nergian	13 14   3A NA   3A A   B C <td< th=""></td<>
Here and the second sec	15 M SA16 SA17 SA10 M SA16 SA17 SA10 M M SA16 SA9 F SA10 M 
	2 VIIIA 8A 2 He Me 10 10 10 10 10 10 10 10 10 10 10 10 10

#### Analytical Chemistry & its importance

The science seeks ever improved meansof measuring the chemical composition of natural and artificial materials by using techniques to identify the substances which may be present in a sample and to determine the exact amounts of the identified substance. Analytical chemistry involves the analysis of matter to determine its composition and the quantity of each kind of matter that is present. Analytical chemists detect traces of toxic chemicals in water and air. They also develop methods to analyze human body fluids for drugs, poisons, and levels of medication.

#### Analytical chemistry can be classified into:

(A) Qualitative analysis which deals with the identification of elements, ions, or compounds present in a sample (tells us what chemicals are present in a sample).

(B) Quantitative analysis which is dealing with the determination of how much of one or more constituents is present (tells how much amounts of chemicals are present in a sample). This analysis can be divided into threemain branches:

(1) Volumetric analysis (Titrimetric analysis): The analyte reacts with a measured volume of the reagent of known concentration, in a process called titration. ( $1^{st}$  grade)

(2) Gravimetric analysis: usually involves the selective separation of the analyte by precipitation, followed by the very non-selective measurement of mass (of the precipitate).  $(2^{nd} \text{ grade})$ 

(3) Instrumental analysis: They are based on the measurement of a physical property of the sample, for example, an electrical property or the absorption of electromagnetic radiation. Examples are spectrophotometry (ultraviolet, visible, or infrared), fluorimetry, atomic spectroscopy (absorption, emission),mass spectrometry, nuclear magnetic resonance spectrometry (NMR), X-ray spectroscopy (absorption, fluorescence). (4<sup>th</sup> grade)

ارشادات مختبرية مهمة

على الطالب قراءة هذه التعليمات والقواعد بدقة والالتزام بها ضمانا ً لسّلامته وسلامة زملائه الطلبة والعاملين معه في المختبر بغية التوصل الى الهدف الاساسي من دخوله المختبر وحصوله على افضل النتائج وأعلى الدرجات وتحقق الاستفادة الفعلية من وقت الحصة المختبرية راجين من طلبتنا الاعزاء عدم مخالفتها أو الاستهانة بها.

1-الالتزام بارتداء الصدرية وذلك حفاظاً على نظافة ملابسك وعدم تلفها وتلوثها بالمحاليل والمواد الكيميائية وارتدائها بداية دخول المختبر.

2– وضع الحقائب و الكتب والسجلات التي تخصك في الاماكن المخصصة لها بعيداءً عن موقع عملك ولا يكون امامك سوى الملزمة المختبرية ودفتر خاص لتسجيل النتائج والملاحظات الخاصة بالتجربة.

- 3- عدم العبث بالاجهزة والادوات التي لا تحتاجها.
- 4- العناية بالنظافة من الصفات الي يجب أن يتحلى بها كل محلل كيميائي.

5- تجنب لمس الجلد أو الانف أو الفم أو العين اثناء العمل الا بعد غسل اليدين بالماء والصابون.

6- عند فتح غطاء لقنينة مادة كيميائية يجب ان يوضع الغطاء بشكل مقلوب على المنضدة لضمان عدم تلوثه وتلوث المنضدة.

7- عدم استعمال ملعقة مادة أو قطارة محلول وادخالها في محلول اخر تجنبا لحدوث تلوث المادة أو المحلول الاخر.

8- عند سحب محلول من قنينة أو اخذ مادة صلبة لا يجوز ارجاع الفائض الى نفس القنينة الاصلية انما تخزن في قنينة اخرى أو تهمل.

9- عند الوزن يجب استعمال قنينة وزن أو دورق صغير نظيف وجاف واحرص على أن يكون الميزان افقياً.

10- عدم استعمال اي اداة زجاجية قبل تنظيفها جيداً وغسلها بالماء المقطر وتجفيفها.

11 – عند سقوط مواد كيميائية على اليد او الملابس يجب غسلها بأكبر كمية من الماء.

### **Qualitative Analysis**

#### Separation of ions to groups and identification

Identification steps at first time to groups by certain reagent and then detection each ion in group:

- 1- Identification of groups by the certain reagent.
- 2- Identification of each ion in the group by a special reagent.

Properties of reagents used in the detection and separation of ions of different groups from each other:

- 1- Abilities to precipitate ions of group which belong to it from the mixture.
- 2- The reagent must be a pure precipitate with the element ions belonging to its group without ions from another group.
- 3- The resulting precipitate must be easily separated from the other ions in solution
- 4- Reagent must be stable, easily to have it and low cost.

### **Analysis of Cations**

Several methods for analysis of cations for metals were used descriptively.

The cations covered in this course will be restricted will include:to thoseof silver, lead, mercury, copper, bismuth, cadmium, arsenic, tin, antimony, iron, manganese, cobalt, nickel, zinc, aluminum, chromium, barium, calcium, strontium, magnesium, sodium, potassium, and ammonium.

The outline will describe the method of precipitating and .analyzing each group. To analyze a general unknown.

Precipitates of group	Precipitation	Ions	Groups
	agent		
			Group I
AgCl, Hg <sub>2</sub> Cl <sub>2</sub> , PbCl <sub>2</sub>	3M HCl	$Ag^{+1}$ , $Pb^{+2}$ , $Hg\frac{+2}{2}$	
HgS, pbS, Bi <sub>2</sub> S <sub>3</sub> , CuS, CdS	H <sub>2</sub> S + 0.3M HCl	IIA= (Cu <sup>+2</sup> , Hg <sup>+2</sup> , Pb <sup>+2</sup> , Bi <sup>+3</sup> , Cd <sup>+2</sup> ) مجموعة النحاس	Group II
$As_2S_3, Sb_2S_3, SnS_2, SnS$		IIB =( $As^{+3}$ , $Sb^{+3}$ , $Sn^{+2}$ ,	
		$\mathrm{Sn}^{+4}$ )	
$Cr(OH)_3$ , $Al(OH)_3$ ,	NH <sub>3</sub> + NH <sub>4</sub> <sup>+1</sup>	مجموعة الزرنيخ IIIA=( Fe <sup>+3</sup> , Cr <sup>+3</sup> ,Al <sup>+3</sup> )	Group III
Fe(OH) <sub>3</sub>		مجموعة الحديد	
MnS, ZnS,NiS,CoS	$H_2S+NH_3+NH_4^{+1}$	IIIB =( Zn <sup>+2</sup> , Mn <sup>+2</sup> , Ni <sup>+2</sup> , Co <sup>+2</sup> ) مجموعة الزنك	
			Group IV
$\begin{array}{c} Ba(PO_{4})_{2,}Sr_{3}(PO_{4})_{2},\\ Ca_{3}(PO_{4})_{2},Mg(NH_{4})PO_{4} \end{array}$	(NH <sub>4</sub> ) <sub>2</sub> HPO <sub>4</sub>	Ba <sup>+2</sup> , Sr <sup>+2</sup> , Mg <sup>+2</sup> Ca <sup>+2</sup> , مجموعة الكالسيوم	
ب خاص بهذه المجموعة حيث	<u> </u> لا یو حد کاشف مر س <i>د</i>	Na <sup>+2</sup> , K <sup>+1</sup> , NH <sup>+1</sup>	Group V
أن لكل ايون كاشفه الخاص به		مجموعة العناصر القلوية	_

### Experiment no (1) Separation and Analysis of First Group (, Pb<sup>+2</sup>Ag<sup>+1</sup>, Hg<sup>+2</sup><sub>2</sub>)

Group I consists of Silver  $Ag^{+1}$ , Lead  $Pb^{+2}$ , and Mercurous(Mercury) Hg  $_{2}^{+2}$  and these ions are common of this group.

The chemical characteristics of the metals to be considered in this course shows that the chlorides of the three ions,  $Ag^{+1}$ ,  $Hg_2^{+2}$  and  $Pb^{+2}$  are insoluble whereas those of the other cations are soluble. It is possible, therefore, to separate these three metals from the others in a general unknown by adding CI<sup>-</sup> to the solution to precipitate the chlorides of lead, silver, and mercurous.

These ions are precipitated by the use of an acid solution of hydrochloric acid at a concentration of (3 M), these precipitations (AgCl, PbCl<sub>2</sub>, and Hg<sub>2</sub>Cl<sub>2</sub>) formed as shown in the equations below:

 $\begin{array}{rcl} Ag^{+1} & + & HCl \rightarrow AgCl \\ Pb^{+2} & + & HCl \rightarrow & PbCl_2 \\ Hg^{+2}_2 & + & HCl \rightarrow & Hg_2Cl_2 \end{array}$ 

## **Procedure:**

1- Transfer 1 ml of the mixtureto test tube then add 3 drops of diluted. HCl (3M).

2- Stir the mixture and put it in the centrifuge (2 min) then separatethe filtrated from the precipitate.

3-Add to the filtrate1 drop of dilutedHCl.

4-The precipitate containsAgCl,PbCl<sub>2</sub>,Hg<sub>2</sub>Cl<sub>2</sub> which are white precipitate.

5- Add 1 ml of hot distilledwater then transfer to water bath (1-2 min).

6- Transfer the test tube to centrifuge while it'shot, separate the filtrated from the precipitate.

7- Each ion will be identified by adding the specific reagent:

A: Add  $K_2Cr_2O_7$  to hot filtrated while contains  $Pb^{+2}$ ,  $Cl^-$  (yellow ppt.).

B: Add (NH<sub>4</sub>OH) to ppt. then dissolve AgCl( black ppt.).

C: Add diluted HNO<sub>3</sub> the precipitate AgCl.

D: dissolve the ppt. of  $Hg_2Cl_2$  in the ( aquaregia ) then add  $SnCl_2$  (white ppt. ) then change to gray after addingan excess of  $SnCl_2$ .



