Unit 1





**Subjects** 

Dear Student After studying this unit you will be able to:

- 1- Understand the importance of analytical chemistry and its applications
- 2- Differentiate between the instrumental and classical analysis and also between qualitative and quantitative analysis .
- 3- Deal with the lab tools , equipment's and chemicals .
- 4- Know the steps of chemical analysis .
- 5-Understand the lab safety and how to act in case of a problem, God forbid .

#### Unit 1

# **INTRODUCTION TO ANALYTICAL CHEMISTRY**

# Introduction



#### **Analytical Chemistry:**







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Is the branch of chemistry that deals with the analysis of substances (analytes) present in the sample qualitatively and quantitatively. In order to accomplish this analysis we must know the physical and chemical properties of these substances.

In other words analytical chemistry deals with the separation ,Identification and determination of substances in a sample .

It also includes coverage of chemical equilibrium and statistical treatment of data .

#### Definitions

sample is anything that comes to mind in the air, water, soil, food and living organisms such as a piece of rock or a piece of meat or some water from the tank of the house or from a river or a lack or a sea or some tissue or blood from humans or animals or some vegetables .... etc. The sample is taken to the laboratory and analyzed for its substances ( analytes) after pretreatment and the final step is the calculations of the percentage of each substance in the sample. An analyte is a constituent of a sample that is analyzed for, and its concentration is determined.



#### Types of analysis (Qualitative Analysis)

Analytical chemistry can be broken down into two general areas of analysis:

Qualitative analysis and Quantitative Analysis and each one of these two types can be either classical or instrumental as we will see .









# Types of analysis (Qualitative Analysis)

#### **Qualitative Classical Analysis:**

This analysis detects (identify) the type of all or some of the substances present in the sample (elements or ions or compounds). In other words it gives an answer to the question : What substances are present in a sample ? these substances can be detected either by a chemical reaction For example, when you add silver nitrate solution to the sample solution a white precipitate formation indicates the presence of chloride ion in the sample.



VIDEO

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VIDEO

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#### **Types of Analysis ( Qualitative Analysis )**

There are also many reagents that give distinctive colors with some of the substances and can be used in the detection of these substances. The flame also can be used for the detection of some common metals.

#### **Qualitative Instrumental Analysis:**

Currently there are many instruments that are separate and distinguish substances (organic or inorganic) in the sample, such as gas chromatography – mass spectrometry (GC - MS), High performance liquid chromatograophy - mass spectrometry (HPLC - MS), infrared spectra (ir) and induced coupled plasma – mass spectrometry (ICP - MS) or ICP - AES (Atomic Emission Spectrometry).



# Types of analysis (Quantitative Analysis)

## **Quantitative Analysis:**

This analysis gives knowledge of the amount of all or some of the substances present in the sample and uses two types of analysis depending on the concentration of the substance in the sample, namely classical chemical analysis and instrumental analysis.



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# Types of analysis (Classical Analysis)



#### **Classical chemical analysis :**

Which depends on the chemical reaction, such as volumetric analysis and gravimetric analysis, as we shall see later in this book . And it uses simple equipment such as burettes , balances , flame , furnace . And is used to estimate high concentrations (more than 0.001 M).

# Types of analysis (Instrumental Analysis)

#### **Instrumental Analysis :**

This type of analysis uses instruments and depends on the physical and physico-chemical properties of the substance being analyzed ( analyte ) such as absorption or emission of electromagnetic radiation (spectroscopic methods of analysis ) or electrical properties of the substance being analyzed such as voltage or current intensity or electrical conductivity ..... etc. (electrochemical methods of analysis) and finally the methods of separation such chromatography.



# Types of analysis (Instrumental Analysis)

All these methods are used to estimate low concentrations of the analyte (less than 0.001 M ), but most of the instrumental analysis methods require expensive instruments.



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#### Main steps in chemical analysis

The analytical process often begins with a question that is not phrased in terms of a chemical analysis. The question could be "Does lead in petrol enter our food supply?" or "Is this water safe to drink?" or "Does emission testing of automobiles reduce air pollution?" A scientist translates such questions into the need for particular measurements. An analytical chemist then must choose or invent a procedure to carry out those measurements.

When the analysis is complete, the analyst must translate the results into terms that can be understood by others preferably by the general public. A most important feature of any results is its limitations. What is the statistical uncertainty in reported results? If you took samples in a different manner, would you obtain the same results? Is a tiny amount (a trace) of analyte found in a sample really there or is it contamination? Once all interested parties understand the results and their limitations, then they can draw conclusions and reach decisions

#### Main steps in chemical analysis (Goal setting)



#### **Goal setting :**

We must set the target of the analysis and how to access it at the lowest cost and fastest time while maintaining good precision (the duplicate results are near to each other ) and good accuracy (the mean of the results of the analysis is very close to the real value).

## Main steps in chemical analysis (Sampling)

#### Sampling :

the sample is taken from something (like water or soil or air, etc.) this sample must represents the average chemical composition of this thing.



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<u>VIDEO</u> You Tube

#### Main steps in chemical analysis (Sampling)

The number of samples taken depends on the homogeneity of this thing example homogeneous air ( few samples ) and a liquid such as water is less homogeneous, so larger number of samples should be taken while the solid - state which is non homogeneous such as soil or rock very larger number of samples must be taken and so on .











#### Main steps in chemical analysis (Drying the sample)

#### **Drying the sample :**

The solid samples usually contain varying amounts of adsorbed water which is a factor can not be controlled therefore we better get rid of it before weighing the sample by drying in the oven and if the analyte is thermally unstable we can use the desicator for drying.







#### Main steps in chemical analysis (Sample Dissolution)

# **Sample dissolution :**

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The chemical reactions are very slow in the solid state compared to in the solution, therefore the sample must be dissolve before analysis. To dissolve the sample we try the water first then dilute acids solutions, hot concentrated acids solutions, mixtures of acids solutions and If the sample does not dissolve we use the fusion where an acidic sample is melted with a base material such as  $Na_2CO_3$  and a basic sample is melted with an acidic substance such as  $K_2S_2O_7$  and if the sample is an oxidant it can be melted with a reductant material and Conversely, where products of the fusion can be dissolved in a water or dilute acid solutions.



#### Main steps in chemical analysis (Sample Dissolution)

Organic compounds are soluble in organic solvents but we should choose the solvent which does not interfere in the measurement process. In the case of estimating inorganic elements in organic samples we must get rid of the organic matter by dry burning ( dry ashing) in the oven or by wet burning (wet ashing) using hot oxidizing concentrated acids such as  $HNO_3$  or  $HCIO_4$  where the organic matter is converted to water vapor and carbon dioxide and therefore we prevent the intervention of organic matter in the analysis of inorganic elements



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Ashing system for the determination of inorganic elements in organic samples

# Main steps in chemical analysis (Storage of Sample Solution)

#### **Storage of Sample Solution :**

During the storage, contamination or loss or disintegration must be avoided. The sample solution must also be stored in a container made from an inert material so that the sample does not interact with it and not be adsorbed on the inner walls of the container. The container must be closed tightly to avoid the affect of the air on the chemical composition of the sample. The sun and heat must be also taken into account because they mayhad an impact on the sample composition.



# Main steps in chemical analysis (Eliminating Interferences)

#### **Interferences :**

An interference is a substance that interfere in the analyte determination for example, cobalt ion Co<sup>2+</sup> can be detected by adding thiocyanate SCN<sup>-</sup>. reagent which reacts with the cobalt to form a red complex, but also iron ion Fe<sup>3+</sup> forms red complex with SCN<sup>-</sup> .Therefore Fe<sup>3+</sup> will interfere in the detection of Co<sup>2+</sup> and must be eliminated by any means before the analysis of  $Co^{2+}$  e.g. by separating iron from cobalt by extraction, precipitation or any other means. Sometimes interferences can be easily eliminated by pH control or by masking agents as we will see later



## Main steps in chemical analysis (Eliminating Interferences)

Specific method of analysis or specific Reagent is a special analysis method for a single substance and can not respond to any other substance on other words , there is no interference . Unfortunately these specific reagents or methods of analysis are practically non-existent . All reagent or methods of analysis are either selective ( respond to a limited number of substances ) and the possibility of the existence of these substances with each other in the sample may be minimal or non-selective ( respond to a large number of substances ) and the possibility of interferences is too high . Unit 1

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# Main steps in chemical analysis (Selecting a method of analysis)

#### Selecting a method of analysis :

There are many methods of analysis to estimate a particular substance in a sample, and to choose one of them depends on several factors such as :

1- **Cost:** Some inexpensive methods of analysis , such as volumetric analysis and gravimetric analysis but most of the instrumental methods of analysis are expensive .

**2-Availability of equipments and materials :** The equipments and the materials of the chosen method of analysis must be available in your laboratory



#### Main steps in chemical analysis (Selecting a method of analysis)

**3-Accuracy and precision needed:** Usually the accurate and precise methods of analysis are expensive. Therefore when for example estimating the salt in sea water there is no need to choose an expensive method because a simple mistake will not result in a disaster but if we analyze mercury in sea water we must choose an accurate and precise method even if it is expensive because mercury is poisonous and any error could result in disaster.





#### Main steps in chemical analysis (Selecting a method of analysis)

4- Sensitivity : The selected method must be able to determine the analyte in the sample , for example if the concentration of the analyte in the sample around 0.00001 M the chosen method must be able to determine 0.00001 M in other words its sensitive should be less than 0.00001 M.

**5- Type of interferences :** We must know the type of impurities existing with the substance to be analyzed in the sample because these impurities or some of which may interfere with the substance to be determined in certain method of analysis and may not interfere in another method . So we choose the method in which the interferences are the least ( more selective ) .

# Main steps in chemical analysis (Selecting a method of analysis)

**6- Speed :** If the analysis requires the results as soon as we must choose a fast method even if it is at the expense of accuracy and precision .

7- The number of samples to be analyzed : If the number of samples to be analyzed is high we must choose an automatic and fast method of analysis . And to choose a suitable method for analyzing a certain substance in a certain sample go back to the folwoing approved journals in analytical chemistry: Analytical Abstract - Analytical Chemistry –Analytica Chimica Acta - Analytical Letters –Analyst - Talanta and many others .

# Main steps in chemical analysis (Measurement)

Measurement : Measure the concentration of analyte in several aliquots. The purpose of replicate measurements (repeated measurements) is to assess the variability (uncertainty) in the analysis and to guard against a gross error in the analysis of a single aliquot.

The uncertainty of a measurement is as important as the measurement itself, because it tells us how reliable a measurement is. If necessary, use different analytical methods on similar samples to make sure that each method gives the same result and that the choice of analytical method is not biasing the result. You may also wish to construct and analyse several different bulk samples to see what variations arise from your sampling procedure.

# Main steps in chemical analysis (Calculating and evaluating results)

#### **Calculating and evaluating the results :**

By knowing the weight of the sample taken for analysis and the amount of analyte in this sample from the results of the analysis, we can calculate the percentage of the analyte in the sample . The results of any chemical analysis must be statistically evaluated otherwise they are not useful .



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In this unit we tried to give a simple definition of analytical chemistry and its significance. Clarify the meaning of sample, analyte and reagent. Demonstrate the difference between the qualitative and quantitative analysis and between classical and instrumental analysis . We briefly discuss the steps of chemical analysis. There are a few basic numerical and experimental tools with which the student must be familiar. .Balances, volumetric flasks, pipets, and ovens are standard pieces of equipment that the student will routinely use in the analytical lab. He should be familiar with the proper way to use these equipment . Finally the student should be aware of lab safety. In this unit, we used some videos and pictures to illustrate some of the concepts of the subject of this unit.

# كتب في الكيمياء التحليلية من تأليف أد ابراهيم زامل عبدالله الزامل

ملخص للمبادئ الأساسية فى الكيمياء التحليلية الدكتور / ابراهيم زامل الزامل - طارق ابراهيم الزامل استاذ الكيمياء التحليلية - بكالوريوس كيمياء قسم الكيمياء - كلية العلوم جامعية الملك سعبود م دار الخبر يجسي للنشر والتوزيع



يناقش هـــذا الكتاب الأسـس والمفاهيم الأساسية في الكيمياء التحليلية والتي لا غنى لأي شخص يعمل في مجال التحليل الكيمائي عنها باسلوب مختصر وواضح مدعم بالأمثلة المحلولة.

المواضيع التي تناولها الكتاب تشمل الحسابات المتعلقة بتحضير المحاليل والعلاقات الكمية في التفاعلات الكيميائية والاتزان الكيميائي مع إعطاء فكرة عن التحليل الحجمي وحساباته وحسابات الـ PH وحاصل الإذابة <sub>sp</sub> كما تناول اشتقاق منحنيات العايرة وكيفية اختيار الدليل المناسب للمعايرة. هذا الكتاب يعطي فكرة مبسطة عن كل من تفاعلات الحموض والقواعد والأكسدة والاختزال والتفاعلات التي تتضمن تكون مركب معقد وتفاعلات الترسيب والتحليل الوزني وكذلك فكرة مبسطة عن تقييم نتائج التحليل إحصائياً. كما زود كل فصل بتمارين وضعت حلولها المضطة في آخر الكتاب .

كتب في الكيمياء التحليلية من تأليف أد ابراهيم زامل عبدالله الزامل

1 al a il التحليل الحجمي والورثي ك الل أدار إيراديسي واصل الزاميل أدار محد فيدالتزيز الحجاجي أجار معد وبدالعزيز الغيرة دارالخريجي للنشر والتوزيع

#### هداالكتاب

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# الاستاذ الدكتور / ابراهيم زامل الزامل التحليك الآلي الطبعة الثالثة الناشر :- دار الخريجي للنشر والتوزيع

#### **الكتاب :** • يناقش هذا الكتاب أغلب ظرق التحليل الآلي الشائعة بأسلوب سهل وواضح حيث يحتوي على ١٨٠ شكلاً توضيحياً وحوالي ٦٠ جدولاً كما يتضمن العديد من الأمثلة المحلولة . ويوجد في نهاية كل فصل أسئلة وتمارين شاملة . كما يوجد في نهاية الكتاب الأجوبة النهائية لبعض

هذه التمارين. ويعالج هذا الكتاب طرق التحليل الطيفي الختلفة مثل الامتصاص الجزئيي والذري والانبعاث الجزيئي والذري وطرق الأشعة السينية وكذلك طرق التحليل الكهربائي مثل الطرق الجهدية والكولوميترية والتوصيلية والفولتاميترية والبولاروجرافية والترسيب الكهربائي كما يناقش طرق الفصل مثل الاستخلاص بالذيبات والتبادل الأيوني والطرق الكروماتوجرافية المختلفة كما يشتمل على معالجة وتقييم نتائج التحليل إحصائيا ويحتوي على ٢٩ تجربة عملية تغطى أغلب الطرق المشروحة. ويوجد في نهاية الكتاب العديد من الملاحق التي تحتوي على الثوابت المهمة في مجال التحليل الآلي وقائمة بالمراجع.

# كتب في الكيمياء التحليلية من تأليف أد ابراهيم زامل عبدالله الزامل

# كتب في الكيمياء التحليلية من تأليف أد ابراهيم زامل عبدالله الزامل



# هذاالكتاب

يعد تلوث البيئة و نتائجه المدمرة من أهم المشاكل التي تواجه البشرية اليوم . و هذا الكتاب يلقى الضؤ على أبعاد هذه المشكلة من الناحية العلمية حيث يتطرق اليأنواع مصادر الطاقة و يوضح علاقة كل منها بـــتلوث البــيئة و يناقــش التركيب الكيميائي الطبيعي لكل من بيئات الهواء و الماء و التربة و الغذاء و التغيرات التي أدخلها الإنسان على هذا التركيب و التي أدت الى تلوث هذه البيئات . و يعالج هذا الكتاب من منظور كيميائي أنواع ملوثات الب\_\_\_\_يئة عامة و مصادرها و أضرارها و طرق تحليلها وسائل مكافحتها كما يتطرق الى مشاكل بيئية عديدة مثل النفايات الصناعية و الأسمدة الكيميائية و المبيدات السامة المسرطنة التي تتواجد في الخضروات و الفواكه التي نأكلها و السموم التي تدخل الى أجسمامنا عبر الأسماك التي نتغذى عليها و كذلك الهرمونات والمضادات الحيوية المتواجدة في اللحوم و المنتجات الحيوانية الأخرى من جراء التعليف و التسمين غير الصحيحين و الأمطار الحمضية و تنقيبة مياه الشرب و معالجة مياه الصرف الصحى . كما يتناول الكتاب مشكلتي نضوب الأوزون و الأنجباس الحراري للأرض اللتين ستدمران الأرض ان لم تتخذ الأجراءات اللازمة . و يحتوي الكتاب على ٣٣ جدولا و ٣٠ شكلا توضيحيا و ٣ ملاحق للمواد الملوثة السمامة و المواد الضارة الحافظة للطعام و العناصر المكونة لجسم الإنسان كما يضم ٩٦ مرجعا عربيا و أجنسا . و يمكن اعتبار هذا الكتاب مرجعا لطلاب و طالبات الجامعات و الكليات و المعاهد و الدورات المتخصصة في علوم البيئة.



# على الراغبين الاستماع الى محاضرات الاستاذ الدكتور/ ابراهيم زامل الزامل باللغة العربية عن هذا الموضوع الرجوع الروابط التالية :

مدخل الى الكيمياء التحليلية

مدخل الى الكيمياء التحليلية ٢