Chemistry Department

Faculty of Science

Umm Al-Qura University

PRACTICAL

Qualitative

Analysis

Introduction to Analytical Chemistry

Everything is made of chemicals. Analytical chemistry determine what and how much. In other words analytical chemistry is concerned with the separation, identification, and determination of the relative amounts of the components making up a sample. Analytical chemistry is concerned with the chemical characterization of matter and the answer to two important questions what is it (qualitative) and how much is it (quantitative).

Analytical chemistry answering for basic questions about a material sample:
· What?
· Where?
· How much?
· What arrangement, structure or form?

Applications of Analytical Chemistry

Analytical chemistry used in many fields:

· In ***medicine***, analytical chemistry is the basis for clinical laboratory tests which help physicians diagnosis disease and chart progress in recovery.

· In ***industry***, analytical chemistry provides the means of testing raw materials and for assuring the quality of finished products whose chemical composition is critical. Many household products, fuels, paints, pharmaceuticals, etc. are analysed by the procedures developed by analytical chemists before being sold to the consumer.

· ***Enviermental quality*** is often evaluated by testing for suspected contaminants using the techniques of analytical chemistry.

· The nutritional value of ***food*** is determined by chemical analysis for major components such as protein and carbohydrates and trace components such as vitamins and minerals. Indeed, even the calories in a food are often calculated from the chemical analysis.

· ***Forensic analysis*** - analysis related to criminology; DNA finger printing, finger print detection; blood analysis.

· ***Bioanalytical chemistry and analysis*** - detection and/or analysis of biological components (i.e., proteins, DNA, RNA, carbohydrates, metabolites, etc.).

Applications of analytical chemistry in pharmacy sciences.

· Pharmaceutical chemistry.

· Pharmaceutical industry (quality control).

· Analytical toxicology is concerned with the detection, identification and measurement of drugs and other foreign compounds (and their metabolites in biological and related specimens.

· Natural products detection, isolation, and structural determination.

Steps in a Chemical Analysis

- Define the problem.
- Select a method.
- Sampling (obtain sample).
- Sample preparation (prepare sample for analysis).
- Perform any necessary chemical separations
- Analysis (perform the measurement).
- Calculate the results and report.

<u>The Language of Analytical Chemistry</u>

Qualitative analysis: An analysis in which we determine the identity of the constituent species in a sample.

Quantitative analysis: An analysis in which we determine how much of a constituent species is present in a sample.

Analytes: The constituents of interest in a sample.

Matrix: All other constituents in a sample except for the analytes.

A **selective reaction** or test is one that can occur with other substances but exhibits a degree of preference for the substance of interest.

A **specific reaction** or test is one that occurs only with the substance of interest.

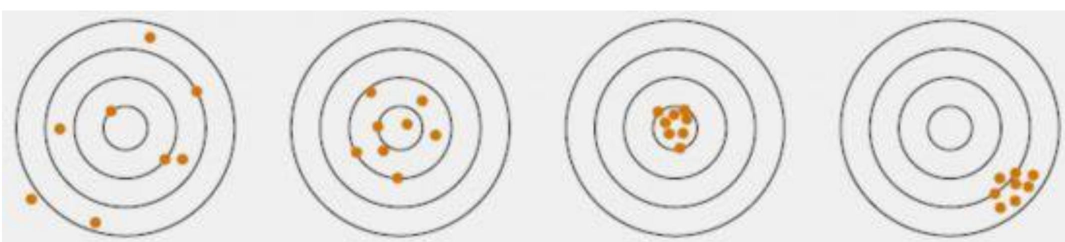
Note: few reactions are specific but many exhibit selectivity.

Detection limit: A statistical statement about the smallest amount of analyte that can be determined with confidence.

Precision and Accuracy

Precision describes the reproducibility of a result. If you measure a quantity several times and the values agree closely with one another, your measurement is precise. If the values vary widely, your measurement is not very precise.

Accuracy describes how close a measured value is to the "true" value. If a known standard is available, accuracy is how close your value is to the known value.



(neither precise nor accurate) (accurate but not precise) (accurate and precise) (precise but not accurate)

Classifying Analytical Techniques

Classical techniques

Mass, volume, and charge are the most common signals for classical techniques, and the corresponding techniques are:

1- Gravimetric techniques.

2- Volumetric techniques.

3- Instrumental techniques.

Instrumental techniques

1- Spectroscopic methods - measuring the interaction between the analyte and electromagnetic radiation (or the production of radiation by an analyte).

2- Electroanalytic methods - measure an electrical property (i.e., potential, current, resistance, amperes, etc.) chemically related to the amount of analyte.

Basic Tools and Operations of Analytical Chemistry

Basic Equipment

Measurements are made using appropriate equipment or instruments. The array of equipment and instrumentation used in analytical chemistry is impressive, ranging from the simple and inexpensive, to the complex and costly.

Equipments for Measuring Mass (Analytical Balance)

An object's mass is measured using a **balance.** The most common type of balance is an in which the balance pan is placed over an electromagnet. Another type of analytical balance is the ***mechanical balances*** which are replaced by the electronic balances.

electronic balance

electronic balance

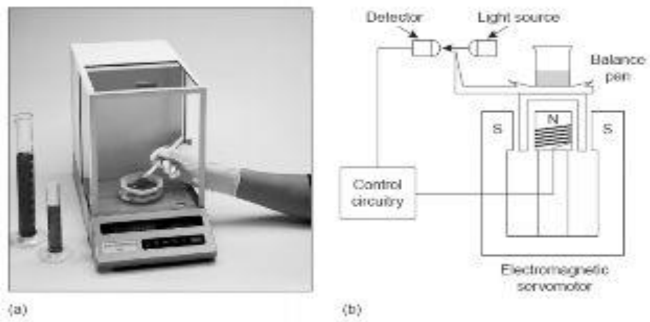


Figure 2.2
(a) Photo of a typical electronic balance.
(b) Schematic diagram of electronic balance; adding a sample moves the balance pan down, allowing more light to reach the detector. The control circuitry directs the electromagnetic servomotor to generate an opposing force, raising the sample up until the original intensity of light at the detector is restored.
Photo courtesy of Fisher Scientific.

Equipment for Measuring Volume

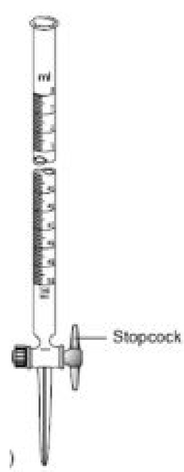
Analytical chemists use a variety of glassware to measure volume. The type of glassware used depends on how exact the volume needs to be.

Volumetric flask is designed to contain a specified volume of solution at a stated temperature, usually 20 °C.

Pipette is used to deliver a specified volume of solution. Several different styles of pipets are available.

Figure 2.5
Common types of pipets and syringes: (a) transfer pipet; (b) measuring pipet; (c) digital pipet; (d) syringe.
Photos courtesy of Fisher Scientific.

Burette is volumetric glassware used to deliver variable, but known volumes of solution. A burette is a long, narrow tube with graduated markings, and a stopcock for dispensing the solution.



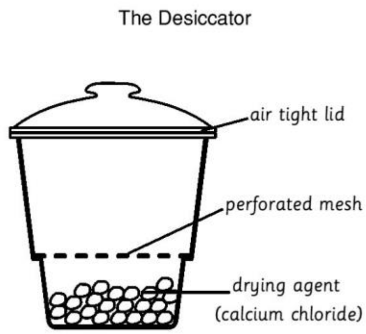
Equipment for Drying

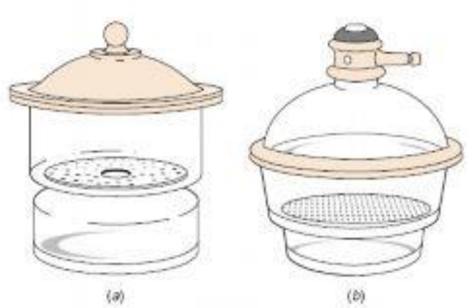
Reagents, precipitates, and glassware are conveniently dried in an oven at 110°C. Many materials need to be dried prior to their analysis to remove residual moisture. Depending on the material, heating to a temperature of 110–140 °C is usually sufficient. Other materials need to be heated to much higher temperatures to initiate thermal decomposition. Both processes can be accomplished using a ***laboratory oven*** capable of providing the required temperature. Commercial laboratory ovens are used when the maximum desired temperature is 160–325 °C (depending on the model). Higher temperatures, up to 1700° C, can be achieved using a muffle ***furnace***.

Conventional laboratory oven used for drying materials.

Example of a muffle furnace used for heating samples to maximum temperatures of 1100–1700 °C.

After drying or decomposing a sample, it should be cooled to room temperature in a desiccator to avoid the readsorption of moisture. A ***desiccator*** is a closed container that isolates the sample from the atmosphere. A drying agent, called a **desiccant,** is placed in the bottom of the container. Typical desiccants include calcium chloride and silica gel.



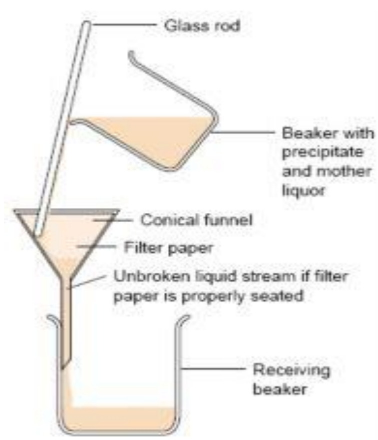


(a) Ordinary desiccator. (*b*) Vacuum desiccator

Filtration

In *gravimetric analysis*, the mass of product from a reaction is measured to determine how much unknown was present. Precipitates from gravimetric analyses are collected by filtration. Liquid from which a substance precipitates or crystallizes is called the **mother liquor**. Liquid that passes through the filter is called **filtrate**.

Filtering a precipitate.
The conical funnel is supported by a metal ring attached to a ring stand, neither of which is shown.



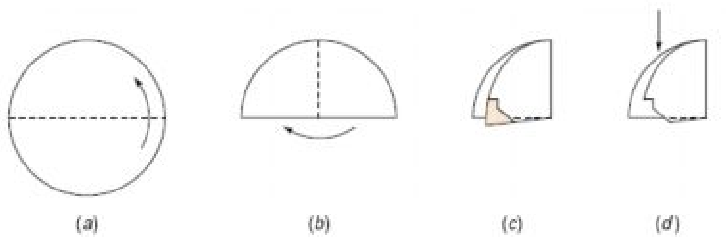
Folding filter paper for a conical funnel.

(*a*) Fold the paper in half.

(*b*) Then fold it in half again.

(*c*) Tear off a corner to allow better seating of the paper in the funnel.

(*d*) Open the side that was not torn when fitting the paper in the funnel.



Preparing Solutions

Preparing a solution of known concentration is perhaps the most common activity in any analytical lab. Two methods for preparing solutions are described in this section.

Preparing Stock Solutions

A ***stock solution*** is prepared by weighing out an appropriate portion of a pure solid or by measuring out an appropriate volume of a pure liquid and diluting to a known volume.

Preparing Solutions by Dilution

Solutions with small concentrations are often prepared by diluting a more concentrated stock solution. A known volume of the stock solution is transferred to a new container and brought to a new volume.

PRINCIPLES FOR SAFETY IN THE CHEMICAL LABORATORY

Safe practices in the chemical laboratory are of prime importance. A student should consider it an essential part of his or her educational experience to develop safe and efficient methods of operation in a lab. To do this, one must acquire a basic knowledge of properties of materials present in the lab, and one should realize the types of hazards that exist and the accidents and injuries that can result from ignorance or irresponsibility on the part of the student or a neighbor.

Regulations

1. W ear safety goggles at all times while in the laboratory.

2. Report all accidents to the instructor or lab assistant immediately.

3. **NEVER** eat, drink, chew, or smoke in the laboratory.

4. **NEVER** leave an experiment unattended. Inform the lab assistant if you must leave the lab.

5. After the experiment is completed, turn all equipment off, making sure it is properly stored, and clean your area.

Failure to comply with these regulations is cause for immediate dismissal from lab.

Precautions

1. Approach the laboratory with a serious awareness of personal responsibility and consideration for others in the lab.

2. Become familiar with the location of safety equipment, such as acid-base neutralizing agents, eye wash, fire extinguisher, emergency shower, and fire blanket.

3. Pay strict attention to all instructions presented by the instructor. If something is not clear, do not hesitate to ask the instructor or lab assistant.

4. Clean up all chemical spills immediately.

5. Be aware of all activities occurring within a reasonable proximity of yourself since you are always subject to the actions of others.

6. To avoid contamination of community supplies, do not use personal equipment such as spatulas in shared chemicals and replace all lids after use.

7. Avoid unnecessary physical contact with chemicals; their toxic properties may result in skin irritation.

8. Use all electrical and heating equipment carefully to prevent shocks and burns.

9. **NEVER** handle broken glassware with your hands; use a broom and a dust pan.

10. Wash your hands at the end of the laboratory.

Personal Attire

Choice of clothing for the laboratory is mainly left to the discretion of the student. Because of the corrosive nature of chemicals, it is in your best interest to wear comfortable, practical clothing. Long, floppy sleeves can easily come into contact with chemicals. A lab coat is suggested to help keep clothes protected and close to the body. Accessories also need consideration. Jewelry can be ruined by contact with chemicals. Open toed shoes do not adequately protect one against chemical spills. If hair is long enough to interfere with motion or observation, it should be tied back. Remember that your clothes are worn to protect you.

Assembling Equipment

Equipment should be assembled in the most secure and convenient manner.

Utility clamps are provided to fasten flasks, etc., to the metal grid work located at the center of each bench. This keeps top-heavy or bulky equipment away from the edge where it can be knocked easily off the bench. Consider the safe location of the hot plate. Keep it near the grid work to minimize chances of contact with the body. If the aspirator is being used, locate your apparatus near the sink for convenience.

Qualitative Analysis for Different Ions

Aim

Training to qualitative analysis of different compounds

Description

Study different experiments to identify compounds by qualitative

Topics:

1- Introduction to analytical chemistry (qualitative analysis) and safety rules
2- Identify acidic radicals of 1st group using dil HCl
3- Identify acidic radicals of 2nd group conc H_2SO_4
4- Identify to miscellaneous group (phosphate, borate, sulphate ions)
5- Identify basic radicals of 1st group such as (lead and silver ions)
6- Identify basic radicals of 2nd group such as (cobber, bithmus, cadmium ions)
7- Identify basic radicals of 3rd group such as (iron and aluminum ions)
8- Identify basic radicals of 4th group such as (Ni^{2+} , Co^{2+}, Zn^{2+}, Mn^{2+})
9- Identify basic radicals of 5th group such as (Ba^{2+}, Ca^{2+} , Sr^{2+})
10- Identify basic radicals of 6th group such as (Na^{+}, NH_4^{+}, K^{+}, Mg^{2+})
11- Revision

- **Reference:**

G. Svehla, "VOGEL'S TEXT BOOK OF MACRO AND SEMIMICRO QUALITATIVE INORGANIC ANALYSIS" 5th edition, Longmane group Limited (1979).

Qualitative Analysis

The separation of the basic radicals into various groups is based on the knowledge of solubility product. For example, in 2nd and 4th groups of basic radicals, the solubility product of sulphides of 2nd group is lesser than 4th group so that the 2nd group need acidic medium and hence only those metal sulphides are precipitated which have low solubility product. On other hand cations in the 4th group need alkaline medium to precipitate.

Anions Radicals are devideds in to three groups

1- **Dilute HCl group:**
   - The main reagent is dil. HCl
   - Anions are carbonate $(CO_3)^{--}$, bicarbonate $(HCO_3)^{-}$ and thiosulphate $(S_2O_3)^{--}$

2- **Concentrated H_2SO_4 group:**
   - The main reagent is conc. H_2SO_4
   - Anions are Cl^-, Br^-, I^-, NO_3^-

3- **Miscellaneous group:**
   - The main reagent is $BaCl_2$
   - Anions are SO_4^{2-}, PO_4^{3-}, $B_2O_4^{2-}$

Cations Radicals are devideds in to six groups

1- **1st group**
   - The main reagent is dil. HCl
   - Cations are Ag^+ and Pb^{2+}

2- **2nd group**
   - The main reagent is dil. HCl + H2S
   - Cations are Cu^{2+}, Bi^{3+}, Cd^{2+}

3- 3rd group

- The main reagent is $NH_4Cl_{(s)}$ + NH_4OH
- Cations are Fe^{3+} , Al^{3+}

4- 4th group

- The main reagent is $NH_4Cl_{(s)}$ + NH_4OH + H_2S
- Cations are Zn^{2+}, Mn^{2+}, Co^{2+}, Ni^{2+}

5- 5th group

- The main reagent is NH_4Cl + NH_4OH + $(NH_4)_2CO_3$
- Cations are Ca^{2+}, Ba^{2+}, Sr^{2+}

6- 6th group

- The main reagent is $Na_2[Co(NO_2)_6]$
- Cations are Na^+, K^+, NH_4^+, Mg^{2+}

Experiment(1)

1- Dil. HCl group

Experments	CO_3^{--}	HCO_3^{-}	$S_2O_3^{--}$
1- Solid salt + dil. HCl	**Eff. with evol. of colorless gas CO_2 which causes turbidity of lime water**	**Eff. with evol. of colorless gas CO_2 which causes turbidity of lime water**	**On warming or standing evol. SO_2 gas and yellowish ppt is formed.**
2- Soln. + $AgNO_3$	**White ppt. soluble in mineral acid**	**White ppt. soluble in mineral acid after heating**	**White ppt soluble in excess $S_2O_3^{--}$and color change from yellow-brown-black**
3- Soln. +$BaCl_2$ or $MgSO_4$ or $CaCl_2$	**White ppt soluble in mineral acids**	**White ppt soluble in mineral acids after boiling**	**3-Soln.+$KMnO_4$ or I_2 give +ve results**

Experiment(2)

2- Conc. H_2SO_4 group

Experiments	Cl^-	Br^-	I^-	NO_3^-
1- Solid+conc.H_2SO_4	**Colourless gas form white clouds with ammonia**	**Brown fumes of Br_2**	**Violet fumes of I_2**	**Faint brown fumes of NO_3, increase by Cu addition.**
2-Soln. + $AgNO_3$	**White ppt sol. In NH_4OH**	**Yellowish-white ppt. sol. In NH_4OH**	**Yellow ppt.**	**2-3ml soln.+1ml of dil H_2SO_4+1ml of freshly $FeSO_4$ soln. $\Downarrow$**
3-Soln. + lead acetate	**White ppt. sol. In hot water**	**White ppt. sol. In hot water**	**Yellow ppt. sol. In hot water**	**No change, by addition conc. H_2SO_4 down the side of tube, brown ring is formed between two layers.**

Experiment (3)

3- Miscellaneous group

Experiment	SO_4^{2-}	PO_4^{3-}	$B_2O_4^{2-}$
1-Soln. + $BaCl_2$	White ppt. insol. In dil. HCl	White ppt. sol. In dil. HCl	White ppt. sol. In dil. HCl
2- Soln. + $AgNO_3$	-ve	Yellow ppt	White ppt. turn to brown by boiling
3-Soln. + Lead acetate	White ppt	-ve	-ve

Experiment(4)

* Cations Radicals

1- First group

Experiment	Pb^{2+}
1- Soln. + dil. HCl	White ppt sol. In boiling water
2- Soln. + dil. H_2SO_4	White ppt.
3- Soln. + K_2CrO_4	Yellow ppt.
4- Soln. + H_2S	Black ppt

Experiment(5)

2- Second group

Experiment	Cu^{2+}	Bi^{3+}	Cd^{2+}
1- Soln. + dil. HCl + H_2S	**Black ppt. sol. In dil. HNO_3**	**Brown ppt. sol. In dil. HNO_3**	**Yellowish ppt. sol. In dil. HNO_3**
2- Soln. + H_2O	**-ve**	**White ppt**	**-ve**
3- Soln. + NH_4OH	**Bluish ppt. dissolve in excess of ammonia formed soluble blue complex**	**White ppt does not soluble in excess of ammonia**	**Soluble colmplex**

Experiment (6)

3-Third group

Experiment	Al^{3+}	Fe^{3+}
1- Soln. + NH_4Cl + NH_4OH	Gel. White ppt. sol. In dil. Acid and base	Reddish brown ppt. sol. In dil. HCl
2- Soln. + NaOH	Gel. white ppt. sol. In excess	Reddish ppt. sol. In dil. HCl
3- Soln. + $K_4[Fe(CN)_6]$	White ppt.	Dark blue ppt.
4- Soln. + KCNS	-ve	Deep red color

Experiment(7)

4-Fourth group

Experiment	Zn^{2+}	Mn^{2+}	Co^{2+}	Ni^{2+}
1- Soln. + NH_4Cl + NH_4OH+ H_2S	White ppt.	Buff ppt.	Black ppt	Black ppt
2- Soln.+NH_4OH	White ppt	Buff ppt.	Blue color	Greenish ppt
3- Soln. + DMG	-ve	-ve	-ve	Red ppt. in presence ammonia
4- Soln. + NH_4CNS	-ve	-ve	Blue color in presence ammonia	
5- Neutral soln. + KNO_3 + acetic acid	-ve	-ve	Yellow ppt	-ve

Experiment (8)

5-Fifth group

Experiment	Ca^{2+}	Ba^{2+}	Sr^{2+}
1- Soln. + $NH_4Cl+NH_4OH+(NH_4)_2CO_3$	**White ppt.**	**White ppt.**	**White ppt.**
2- Soln. + K_2CrO_4	**-ve**	**Yellow ppt.**	**-ve**
3- Soln. + $CaSO_4$	**-ve**	**White ppt.**	**Cloudy soln. after heating**
4- Flame test	**Brick red color**	**Yellowish green color**	**Crimson red color**

Experiment(9)

6-Six group

Experiment	Na^+	K^+	NH_4^+	Mg^{2+}
1- Soln. + $Na_2[Co(NO_2)_6]$	-ve	Yellow ppt.	Yellow ppt.	Yellow ppt.
2- Soln. + NaOH	-ve	-ve	-ve After heating, ammonia odor is observed	White ppt.
3- Flame test	Golden yellow color	Violet color	-ve	-ve

Report

Physical properties:

1- **Color:**

2- **Shape:**

3- **Solubility:**

Chemical properties:

Experiments	Observation	Results

Report

Physical properties:

1- **Color:**

2- **Shape:**

3- **Solubility:**

Chemical properties:

Experiments	Observation	Results

Report

Physical properties:

1- **Color:**

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Chemical properties:

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Physical properties:

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Chemical properties:

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Report

Physical properties:

1- **Color:**

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Chemical properties:

Experiments	Observation	Results