

# SEMESTER V

Course Code: CHE5B06

Core Course VI:

**INORGANIC CHEMISTRY – III**

# SYLLABUS

**Module I: Analytical Principles II :** Applications of solubility product and common ion effect in the precipitation of cations – Interfering acid radicals and their elimination (oxalate, fluoride, borate, phosphate, chromate, arsenite and arsenate) – Introduction of micro scale experiments in inorganic and organic qualitative analysis & their advantages. Preparation of  $\text{Na}_2\text{CO}_3$  extract for inorganic qualitative analysis and its advantages. Gravimetric analysis – Mechanism of precipitate formation. Factors affecting stability of precipitates. Co-precipitation and post precipitation. Effects of digestion, washing, drying and ignition of precipitates.

**Module VI: Environmental Pollution :** Air pollution: Major air pollutants – Oxides of carbon, nitrogen and sulphur – Particulates – London smog and photochemical smog. Effects of air pollution: Acid rain, greenhouse effect and depletion of ozone. Control of air pollution – Alternate refrigerants. Bhopal Tragedy (a brief study). Water pollution: Water pollution due to sewage and domestic wastes – Industrial effluents – Agricultural discharge – Eutrophication. Quality of drinking water – Indian standard and WHO standard. Water quality parameters: DO, BOD and COD – Determination of BOD and COD. Toxic metals in water (Pb, Cd and Hg) – Minamata disaster (a brief study). Control of water pollution – Need for the protection of water bodies. Thermal pollution, noise pollution and radioactive pollution (Sources, effects and consequences). Pollution due to light. Hiroshima, Nagasaki and Chernobyl accidents (a brief study). Local environmental movements: Silent Valley, Plachimada, Narmada. Air pollution in Indian cities (Delhi, Agra and Kanpur).

# Micro analysis

## Macro Analysis

Analyte masses are  
greater than 0.1 g.

## Semimicro analysis:

Analyte in the range of 0.1 to 0.01 g.

## Micro analysis:

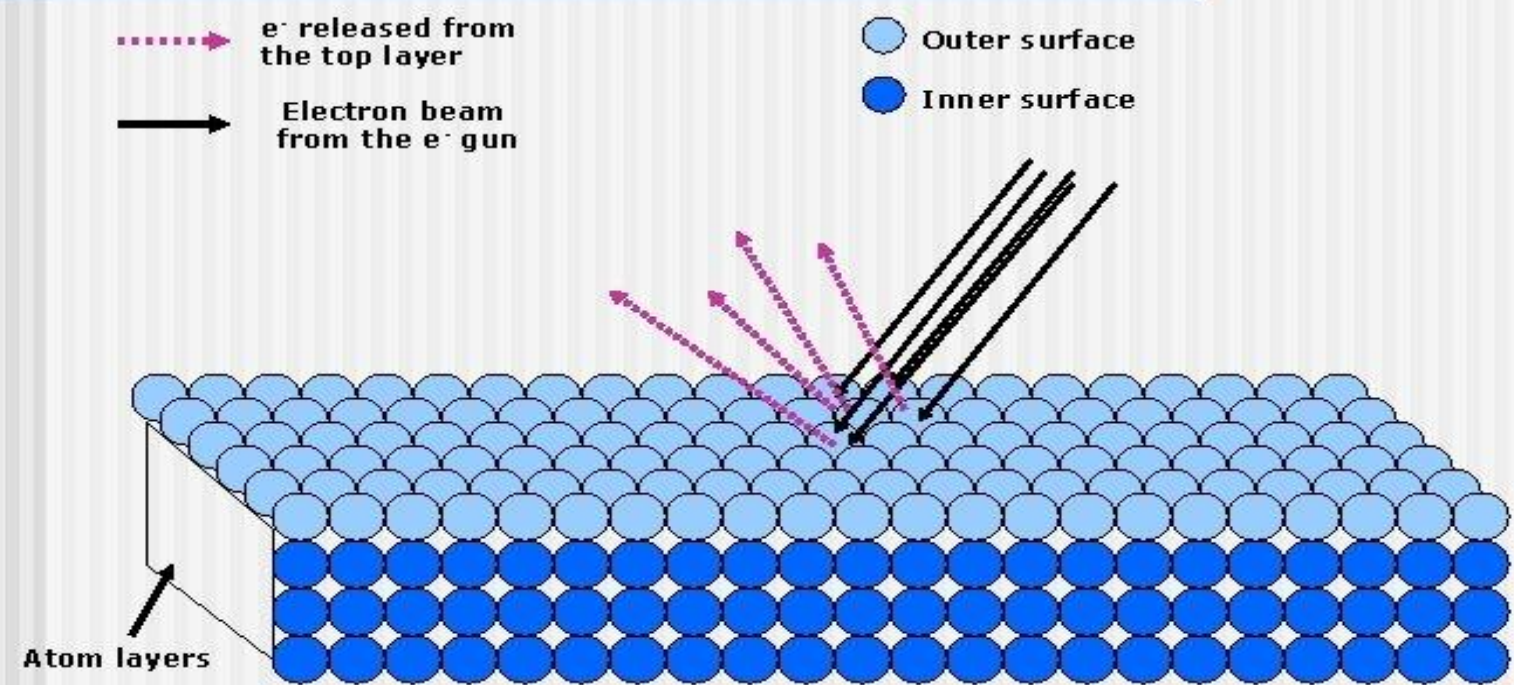
0.01 g to 0.0001g.

## Ultramicro analysis:

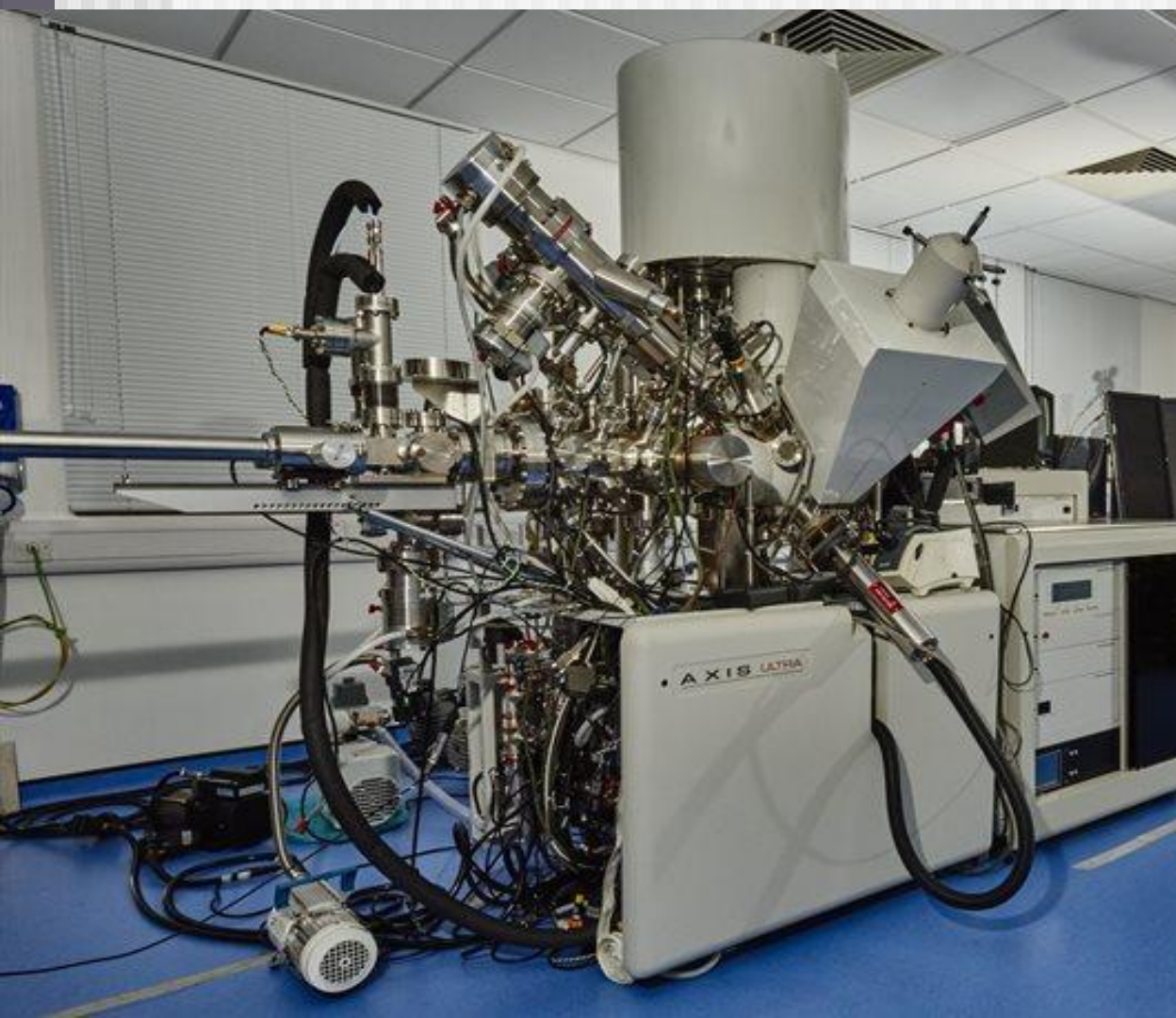
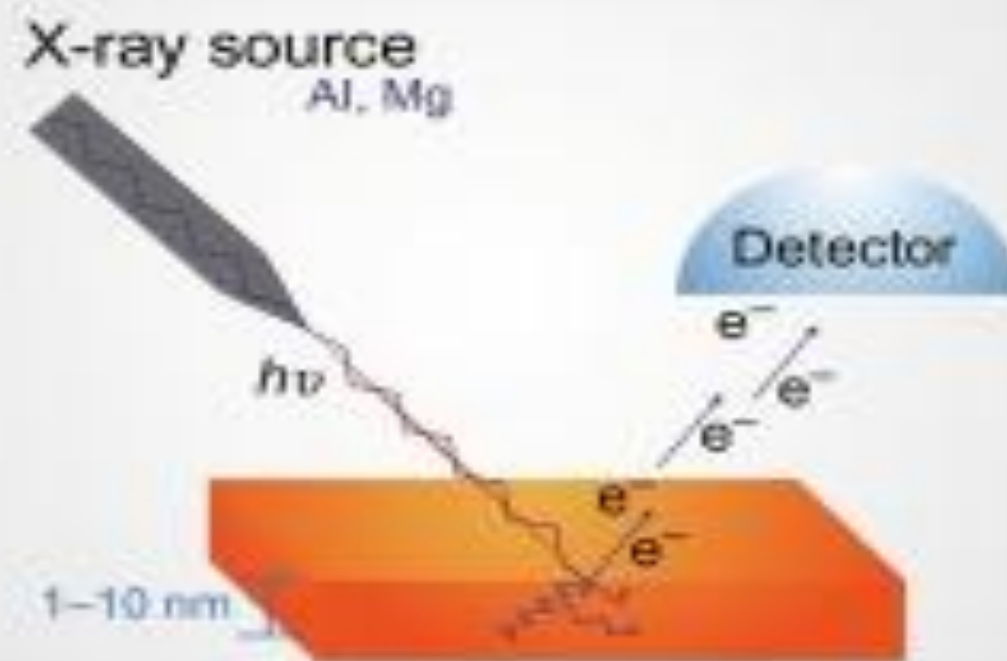
Less than 0.0001 g



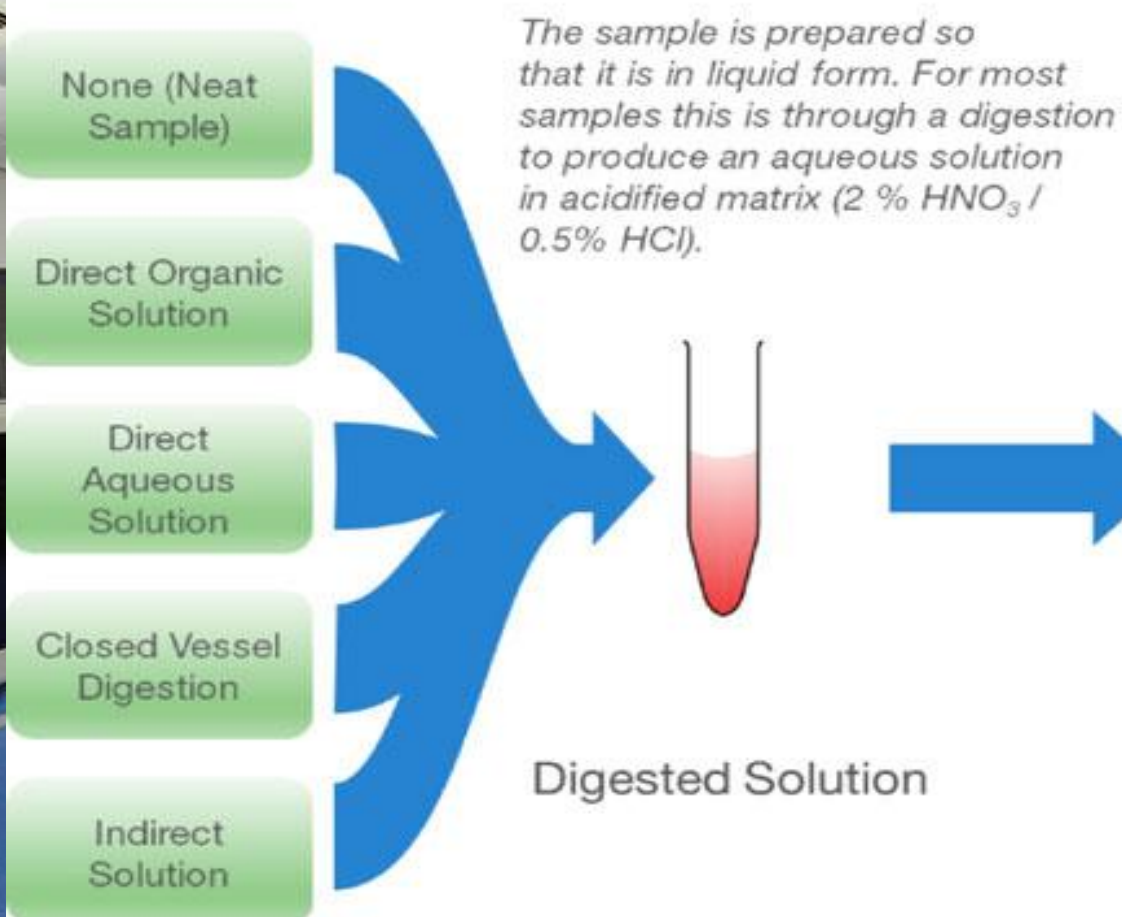
# Auger Electron Spectroscopy (AES)



# X-ray photoelectron spectroscopy



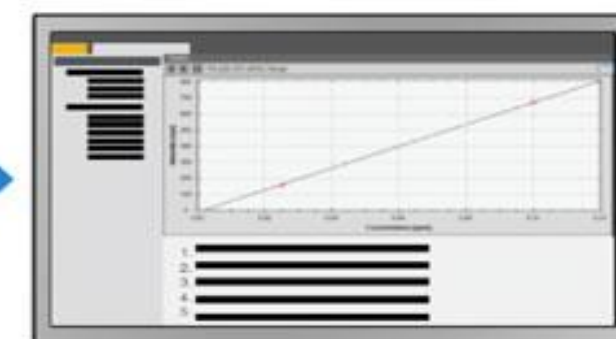
## Sample Preparation



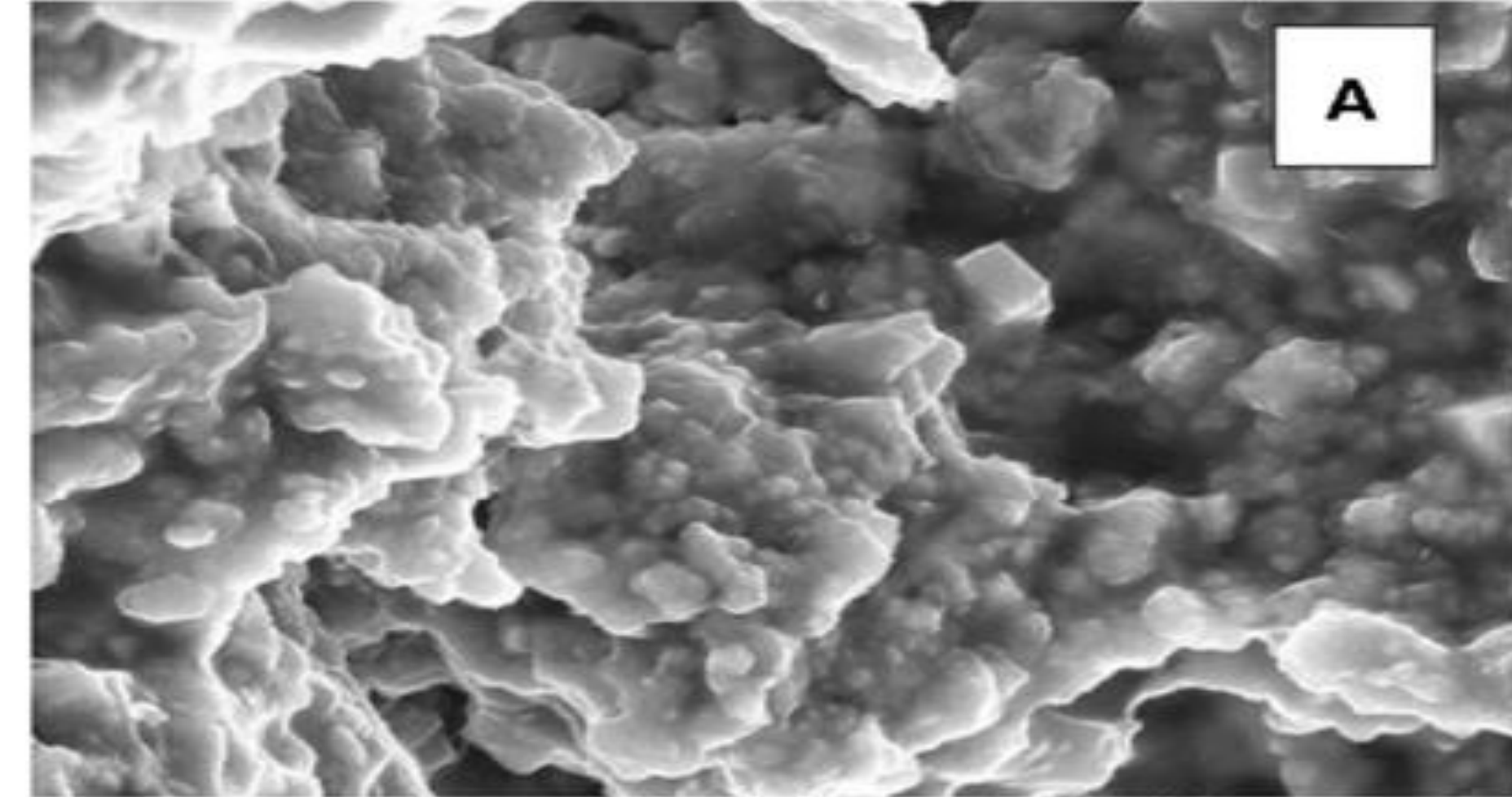
## Sample Analysis



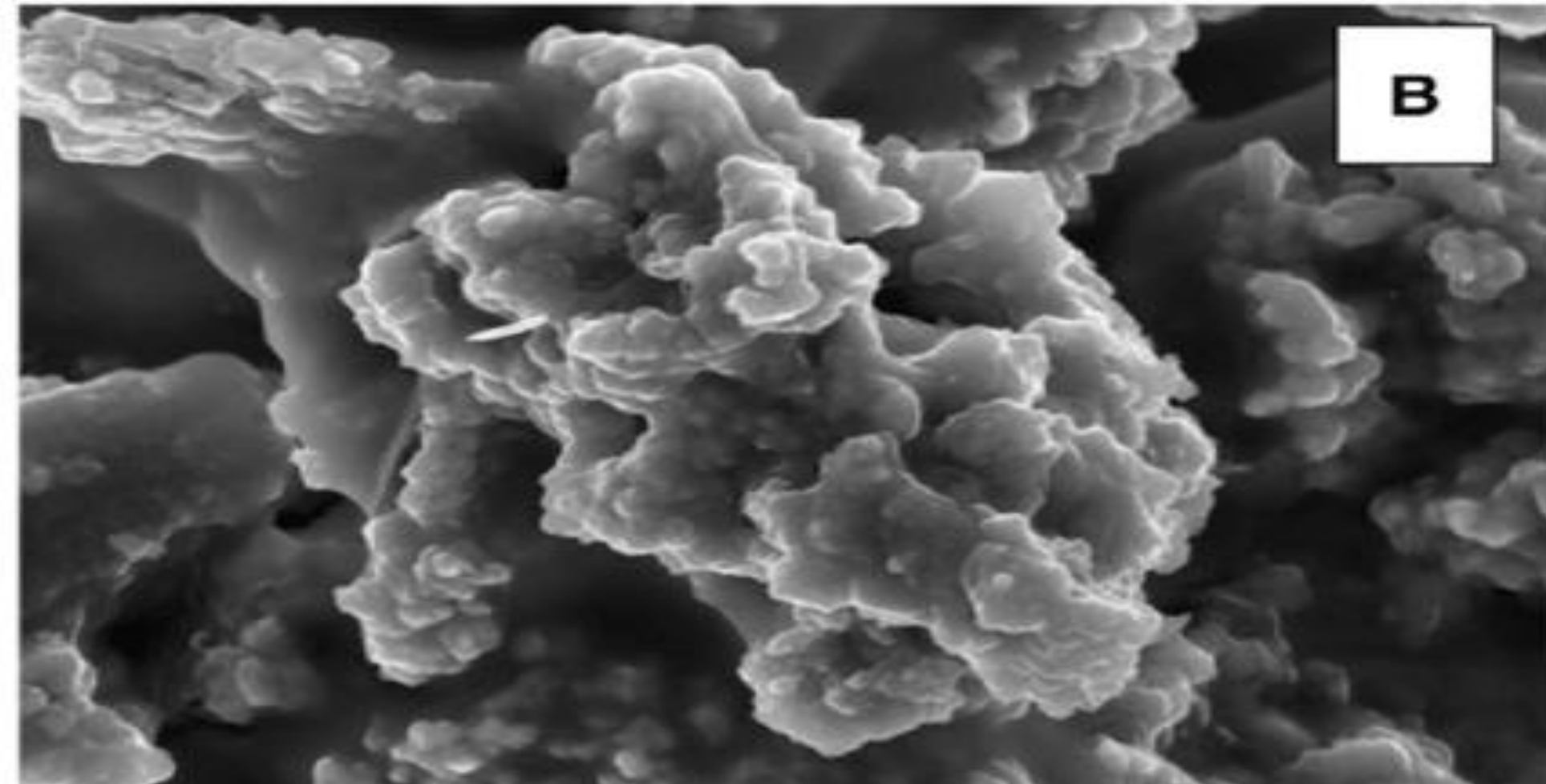
## Data Analysis



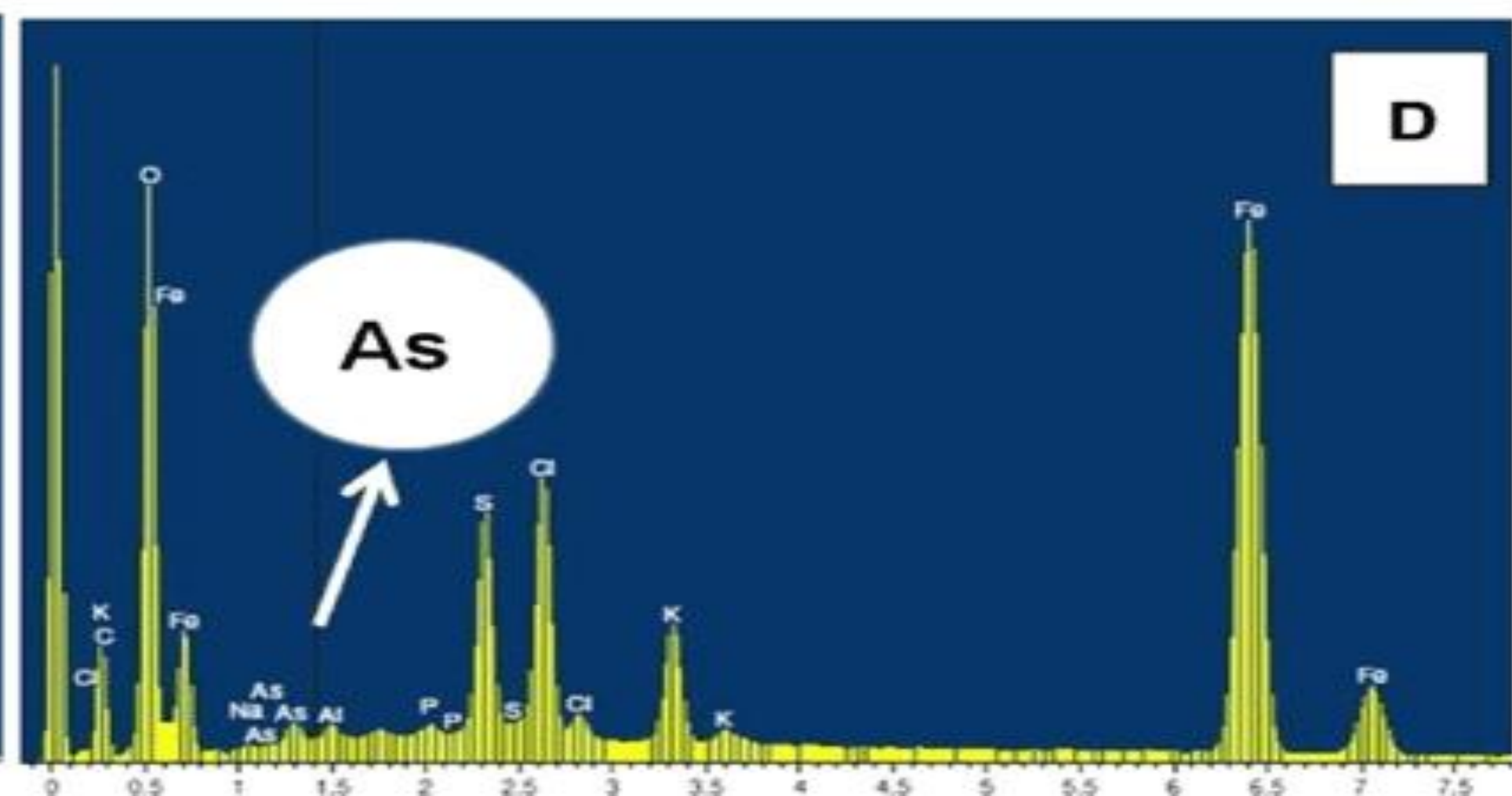
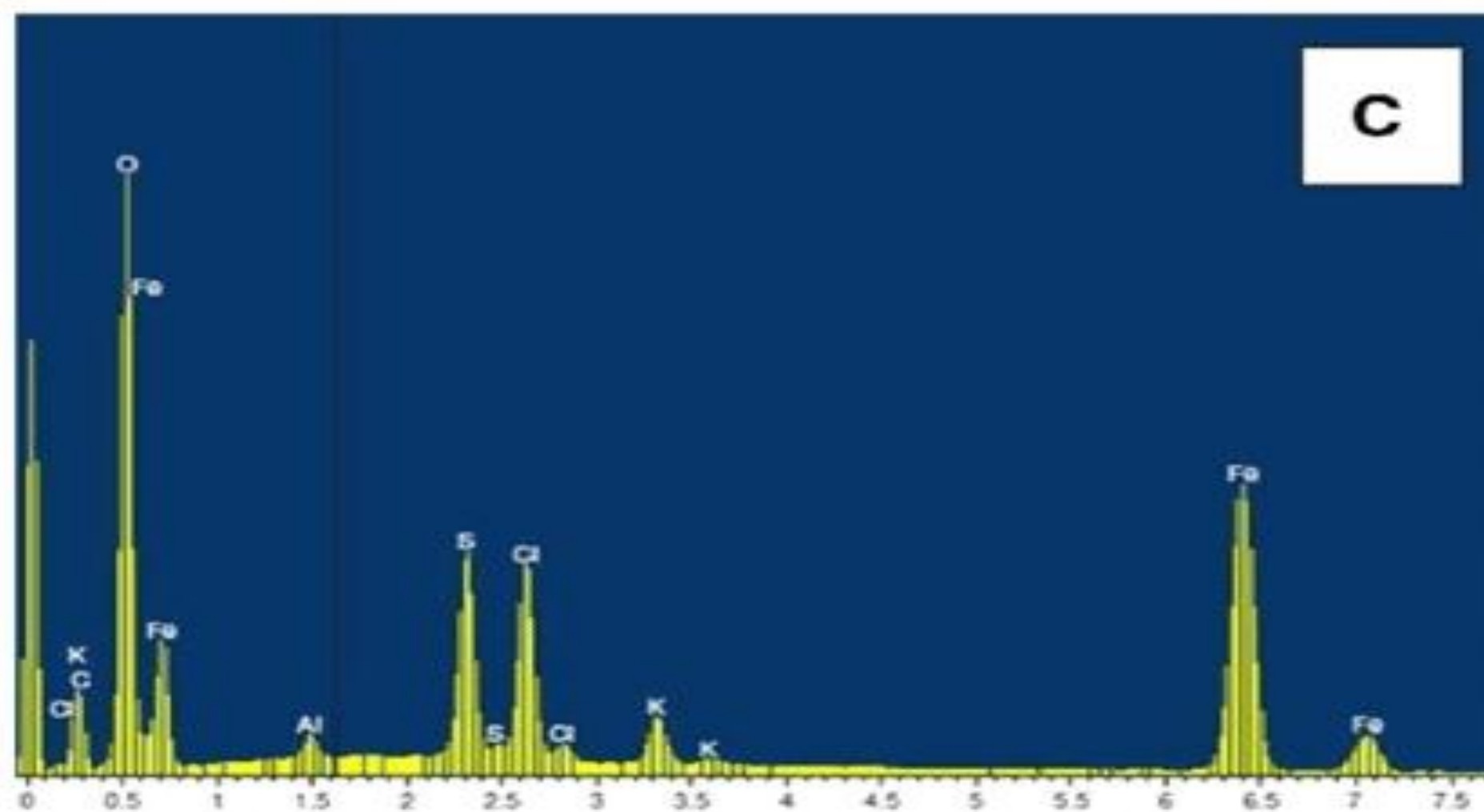




1  $\mu$ m  
EHT = 10.00 kV  
WD = 5.2 mm  
Signal A = InLens  
Mag = 30.00 K X  
Date :20 Jul 2017  
Time :15:02:27



1  $\mu$ m  
EHT = 10.00 kV  
WD = 5.0 mm  
Signal A = InLens  
Mag = 30.00 K X  
Date :20 Jul 2017  
Time :15:22:07



# Micro analysis

- ❖ Centrifuge tubes of 0.5 to 2 mL capacity for most operations in a microanalysis.

*Solutions are almost always separated from precipitates by centrifuging either manually or electrically). After centrifuging a system containing a precipitate, the supernatant liquid is usually removed either by a capillary dropper or a transfer capillary pipette or a capillary siphon. After removal of the supernatant solution, the precipitate remaining in a micro centrifuge tube can be washed by adding the wash solution directly to the precipitate and then stirring the system with a thin glass rod*

- ❖ If strong heating of solutions is required *micro beakers (5 mL) or micro crucibles (2 mL) are used*
- ❖ The passage of a gas like  $H_2S$  into a solution in a micro centrifuge tube is carried out by leading the gas through a fine capillary tube.
- ❖ heated on a metal hot plate or on micro burners. *Digestion of solid samples with acid or solvent is performed in small porcelain or silica crucibles*
- ❖ Small *silica watch glasses* find application for evaporations.
- ❖ Fusion of a small solid sample is conducted on a platinum wire loop or in a platinum spoon (0.5–1 mL capacity).





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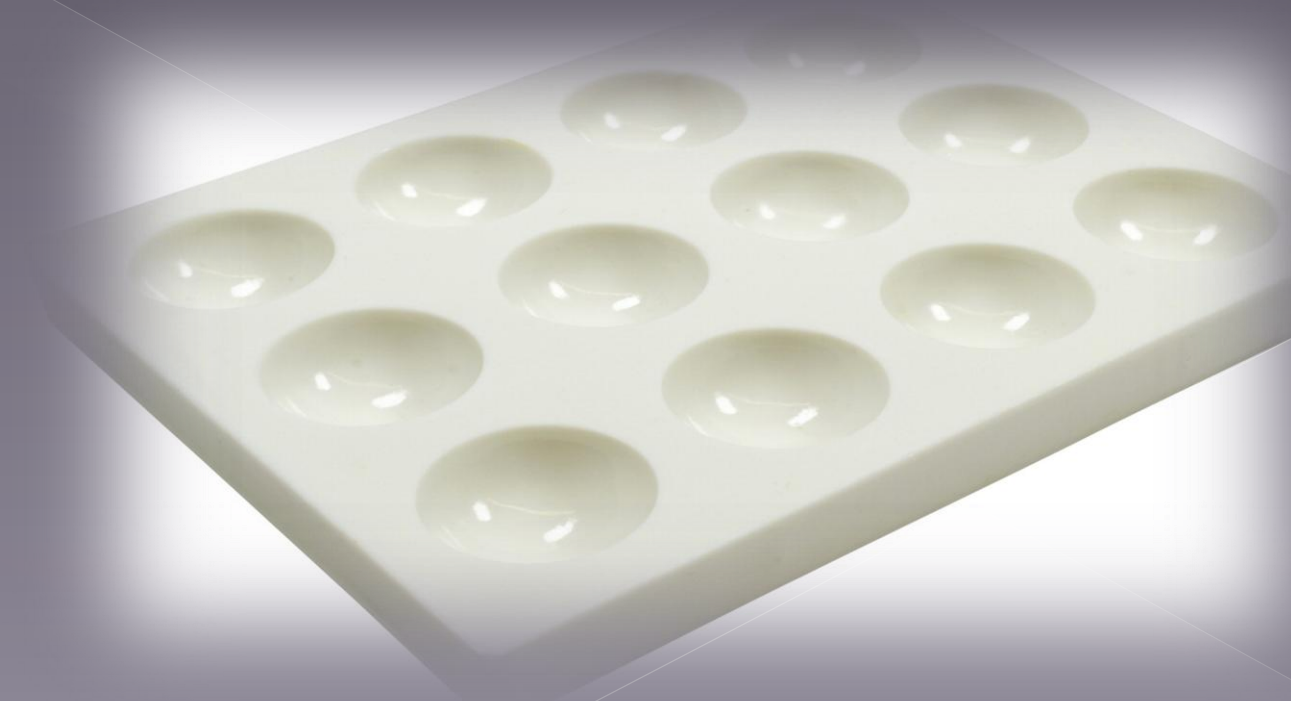
# Spot Test

The term spot test or spot reaction refers to a micro test for ions or compounds, generally used in a micro scale inorganic or organic qualitative analysis

Such a chemical test involves manipulation *with very small quantities of analyte sample and reagents*—a drop or two of solutions or liquid compounds, or a crystal or two of solids.

- 1. by bringing together a drop of the test solution and a drop of the reagent on a glass, porcelain or paper surface, e.g., a glazed porcelain “spot plate” with or without small depressions*
- 2. by placing a drop of the analyte solution on a filter paper impregnated with the necessary reagent, and by bringing into contact a drop of the reagent or a strip of reagent paper with the gases liberated from minute quantities of a sample in a micro test tube or centrifuge tube<sup>1</sup>.*

Spot test for Fe(III): Place a drop of the test solution on a spot plate and add 1 drop of 1 % ammonium thiocyanate solution. A deep red (“blood red”) colouration due to the formation of iron(III) thiocyanate appears if Fe<sup>3+</sup> is





# Advantages of micro analytical methods

- ❑ Micro analysis requires only a very small quantity of the sample.
- ❑ Very small quantities of solvent and reagent are utilized- less waste and more cost effective -less pollution-saves a lot of money.
- ❑ Greater sensitivity, selectivity and specificity.
- ❑ Possible to avoid the special separations and operations such as filtration and dissolution of precipitates
- ❑ Spot tests diminish sources of error
- ❑ Needs short time.
- ❑ Small equipment's-easy handling are much cheaper
- ❑ Less storage place-Less current
- ❑ Pleasant working atmosphere