

### 5.3 CHEMICAL ANALYSIS

- ❖ Chemical analysis is used to identify the contents, composition and quality of the materials used in product development, manufacturing and testing.

#### 5.3.1 CHEMICAL PROPERTY

- ❖ A chemical property is a characteristic or behavior of a substance that may be observed when it undergoes a chemical change or reaction.

S.No	Property	Description
1.	Toxicity	Toxicity is a very important chemical property because it gives the harm of a substance can bring to other organisms.
2.	Reactivity	Reactivity is the tendency of a substance to undergo chemical reaction, either by itself or with other materials, and to release energy.
3.	Types of chemical bonds formed	Chemical bonds include covalent, polar covalent and ionic bonds
4.	Coordination number	The coordination number of an atom in a molecule is the number of atoms bonded to the atom, the coordination number describes the number of neighbour atoms with respect to a central atom
5.	Oxidation states	The oxidation state is the charge of an atom if all bonds it formed were ionic bands.
6.	Flammability	Flammable is a property of a material relating how easily the material ignites or sustains a combustion reaction
7.	Heat of combustion	A combustion reaction involves oxygen and releases energy as heat.

8.	Enthalpy of formation	The heat of formation is the heat released or absorbed (enthalpy change) during the formation of a pure substance from its elements at constant pressure (in their standard states). Heat of formation is also called enthalpy of formation kilojoules per mole (kJ/mol).
9.	Chemical stability under specific conditions	Stability occurs when a system is in its lowest energy state, or chemical equilibrium with its environment
10.	Acidity or basicity	Acidity is the extent to which a substance will donate a proton/hydrogen ion. Basicity is the extent to which a substance will accept a proton/hydrogen ion.
11.	Radioactivity	Radioactive decay is a property of several naturally occurring elements as well as of artificially produced isotopes of the elements

### **5.3.2 CHEMICAL TESTING**

- ❖ Chemical Testing provides a variety of quantitative and qualitative services for verification, identification and component analysis of ferrous and non-ferrous metals.

### **5.3.3 PURPOSE OF CHEMICAL TESTING**

- ❖ Chemical Trace Analysis
- ❖ Elemental Trace Analysis
- ❖ Failure Analysis
- ❖ Contamination Analysis
- ❖ Materials Analysis and Testing
- ❖ Material Verification
- ❖ Material Identification
- ❖ Chemical Composition Analysis.

### **5.3.4 CHEMICAL COMPOSITION TECHNIQUE AND TESTS**

#### **(a) Chromatography Technique**

- ❖ Gas Chromatography
- ❖ Ion Chromatography
- ❖ Liquid Chromatography

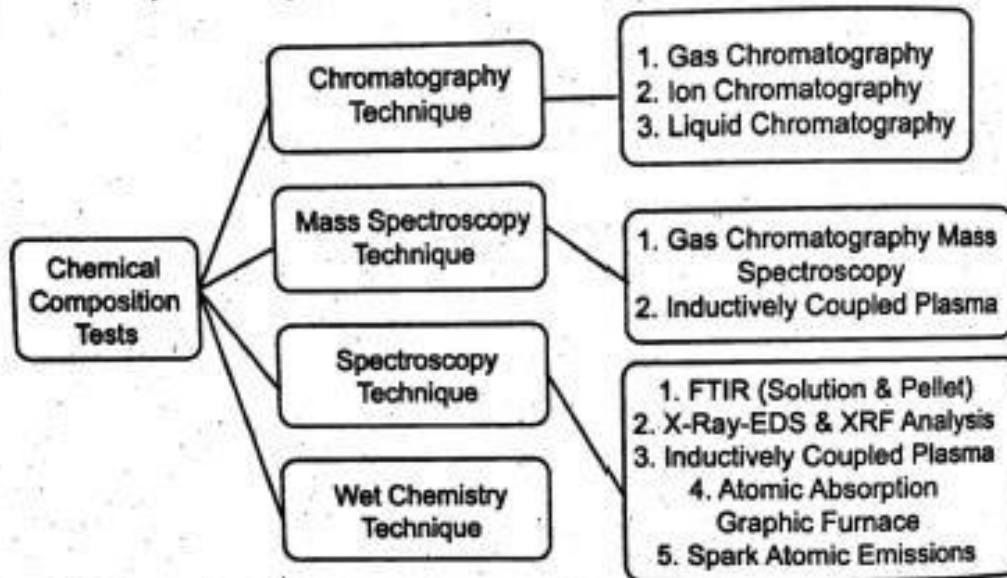
#### **(b) Mass Spectroscopy Technique**

- ❖ Gas Chromatography Mass Spectroscopy
- ❖ Inductively Coupled Plasma

#### **(c) Spectroscopy Technique**

- ❖ Fourier-transform infrared spectroscopy (Solution & Pallet)
- ❖ X-Ray-EDS & XR F Analysis
- ❖ Inductively Coupled Plasma (ICP-AES)
- ❖ Atomic Absorption Graphite Furnace (GF-AAS)
- ❖ Spark Atomic Emissions (Spark-AES)

#### (d) Wet Chemistry Technique



*Fig. 5.8. Types of chemical composition test*

#### 5.3.5 X-RAY FLUORESCENCE

- ❖ XRF (X-ray fluorescence) is a non-destructive analytical technique used to determine the elemental composition of materials. XRF analyzers determine the chemistry of a sample by measuring the fluorescent X-ray emitted from a sample when it is excited by a primary X-ray source.
- ❖ The phenomenon is widely used for elemental analysis and chemical analysis, particularly in the investigation of metals, glass, ceramics and building materials, and for research in geochemistry,

##### 1. PRINCIPLE

- ❖ **X-ray fluorescence (XRF)** is the emission of characteristic "secondary" (or fluorescent) X-rays from a material that has been excited by being bombarded with high-energy X-rays or gamma rays.

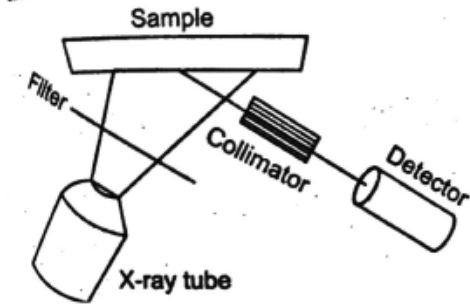
## 2. COMPONENTS OF A TYPICAL XRF SPECTROMETER

- ❖ Source of X-rays used to irradiate the sample. Wavelengths are typically in the range 0.01 to 10 nm, which is equivalent to energies of 125 keV to 0.125 keV.
- ❖ Detection equipped by Gas-filled detectors, semiconductor detector, scintillation detector, a photographic plate.

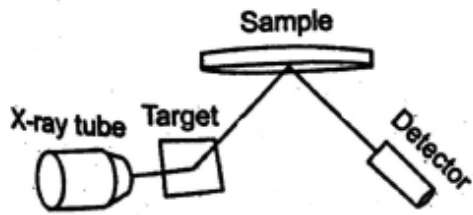
## 3. TYPES OF XRF SPECTROSCOPY.

The XRF spectroscopy differs primarily by detection and analysing.

- ❖ Energy Dispersive XRF (Direct and polarized excitation)
- ❖ Wavelength Dispersive XRF

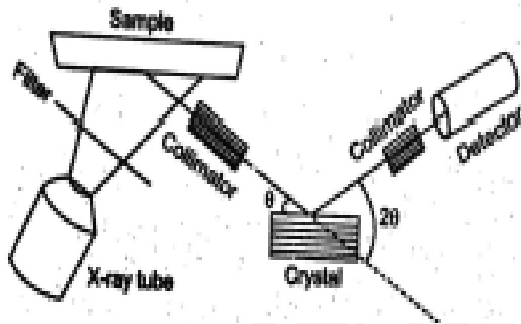
Methods	Description
<p><b>Energy Dispersive X-ray fluorescence with direct excitation</b></p>  <p>The diagram illustrates the setup for Energy Dispersive X-ray fluorescence with direct excitation. It shows an X-ray tube on the left, which emits X-rays through a filter. These X-rays strike a sample, causing it to fluoresce. The emitted fluorescence passes through a collimator and is then detected by a detector on the right.</p>	<p>An energy dispersive detection system directly measures the different energies of the emitted X rays from the sample. By counting and plotting the relative numbers of X-rays at each energy an XRF spectrum is generated</p>

### Energy Dispersive X-ray fluorescence with polarized excitation



The detector must be perpendicular to the plane determined by the tube, target and sample. The most important effect is that by deflecting the X-ray radiation by  $90^\circ$ , the radiation is polarized and the spectral background in the spectrum is reduced.

### Wavelength Dispersive X-ray fluorescence



The X-rays are directed to a crystal, which diffracts the X-rays in different directions according to their wavelengths (energies). On a sequential system a detector is placed at a fixed position, and the crystal is rotated so that different wavelengths are picked up by the detector.

## 4. WORKING OF X-RAY FLUORESCENCE

1. A solid or a liquid sample is irradiated with high energy X-rays from controlled X-ray tube.
2. When an atom in the sample is struck with an X-ray of sufficient energy, an electron from one of the atom's inner orbital shells is removed.
3. The atom regains stability, filling the vacancy left in the inner orbital shell with an electron from one of the atom's higher energy orbital shells.
4. The electron drops to the lower energy state by releasing a fluorescent X-ray. The energy of this X-ray is equal to the specific difference in energy between two quantum states of the electron. The measurement of this energy is the basis of XRF analysis.

5. The intensity of each characteristic radiation is directly related to the amount of each element in the material.

## 5. APPLICATIONS

- ❖ It is a method of elemental (metal and Nonmetal) analysis with atomic number greater than 12.
- ❖ Quantitative analysis can be carried out by measuring the intensity of fluorescence at the wavelength characteristics of the element being determined, especially applicable to most of the element in the periodic table.
- ❖ Research in igneous, sedimentary, and metamorphic petrology, Soil surveys, Mining (e.g., measuring the grade of ore), Cement production, Ceramic and glass manufacturing
- ❖ Metallurgy (e.g., quality control)
- ❖ Environmental studies (e.g., analyses of particulate matter on air filters)
- ❖ Petroleum industry (e.g., sulphur content of crude oils and petroleum products)
- ❖ Field analysis in geological and environmental studies (using portable, hand-held XRF spectrometers)
- ❖ Bulk chemical analyses of major elements and trace elements

## 6. ADVANTAGES

- ❖ Simple spectra analysis
- ❖ XRF is a versatile and rapid technique
- ❖ Easily analysis of the element among the same family elements
- ❖ It is non-destructive method of chemical analysis
- ❖ Important as in case of samples in limited amounts, or valuable or irreplaceable
- ❖ It is precise and with skilled operations it is accurate
- ❖ Applicable to a wide variety of samples from powders to liquids
- ❖ It is convenient and economical to use
- ❖ The instruments have few moving parts, tend to be low-maintenance, and on a regular basis consume only liquid nitrogen and electricity
- ❖ Spectral positions are almost independent of the chemical state of the analyses

- ❖ Applicable over a wide range of concentrations

## 7. DISADVANTAGES

- ❖ It fairly high limits of detection when compared to other methods
- ❖ Possibility of matrix effects, although these can usually be accounted for using software-based correction procedures
- ❖ It is limited in their ability to precisely and accurately measure the abundances of elements with atomic number  $< 11$  in most natural earth materials
- ❖ XRF analyses cannot distinguish variations among isotopes of an element
- ❖ XRF analyses cannot distinguish ions of the same element in valence states
- ❖ Instrumentation is fairly expensive

