MODULE 5

Chemical imaging techniques and analytical tools

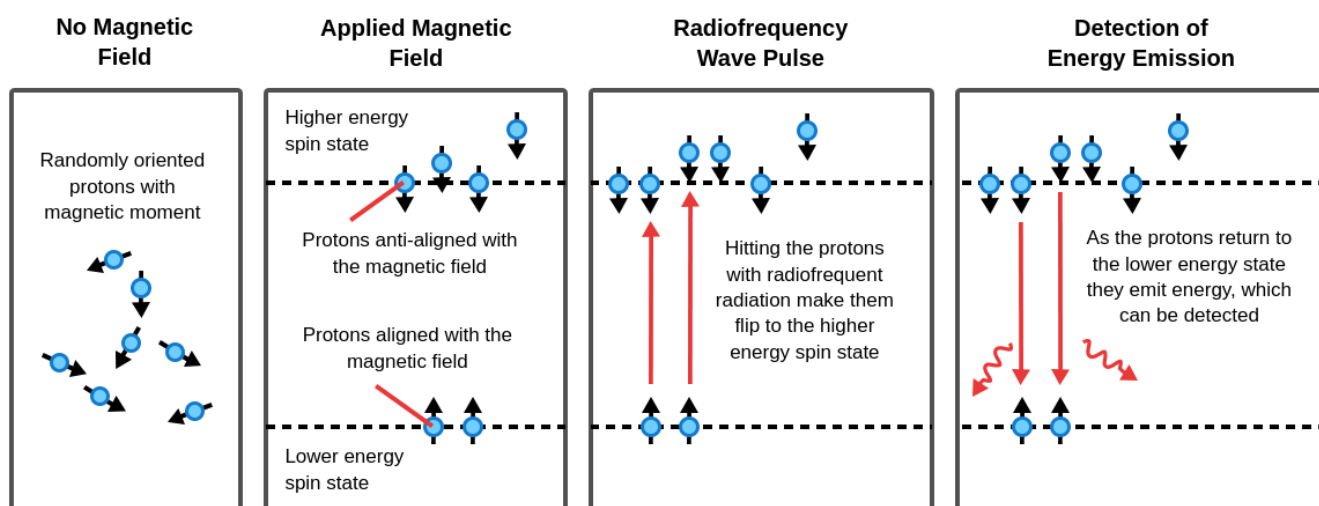2. **Discuss the principle and application of NMR spectroscopy in imaging.**
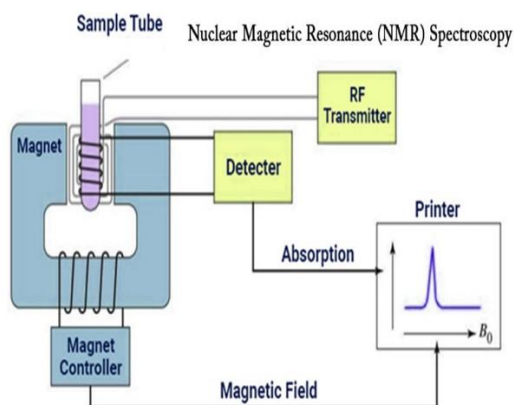
OR

Illustrate the spectroscopic technique used in MRI Scanning.

NMR uses magnetic properties of certain nuclei and interaction of nuclei of an atom and radiofrequency waves of 4 to 900 MHz produces a spectrum.

Principle: NMR spectroscopy is applicable only to nuclei with odd spin such as 1_H and 13_C

No Magnetic Field	Applied Magnetic Field	Radiofrequency Wave Pulse	Detection of Energy Emission
Randomly oriented protons with magnetic moment	Higher energy spin state — Protons anti-aligned with the magnetic field; Protons aligned with the magnetic field; Lower energy spin state	Hitting the protons with radiofrequent radiation make them flip to the higher energy spin state	As the protons return to the lower energy state they emit energy, which can be detected

Instrumentation

Sample Tube — Nuclear Magnetic Resonance (NMR) Spectroscopy
RF Transmitter
Magnet
Detecter
Printer
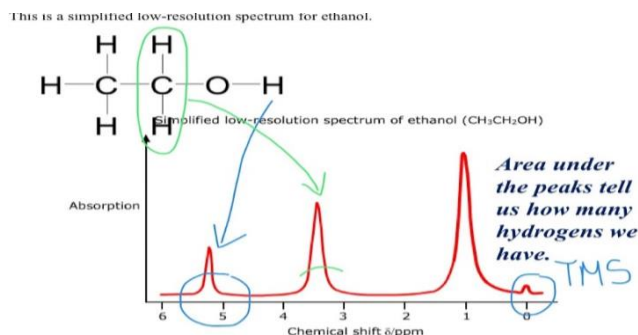Absorption
Magnet Controller
Magnetic Field

Working: The sample is placed in a powerful magnet, which causes the nuclei in the sample to align with the field. Radio waves are used to excite the nuclei and cause them to absorb energy. This absorption occurs when the frequency of the radio waves matches the frequency of precession of the nuclei. Sensitive radio receivers detect the NMR signals produced by the nuclei. The data is processed and recorded in a computer to produce an NMR spectrum. The spectrum gives the chemical structure of a molecule.

Application: Intensity of absorption is related to number of equivalent protons.

Position of the spectral line is related to the type of chemical environment (ref TMS).

Example:

This is a simplified low-resolution spectrum for ethanol.

Simplified low-resolution spectrum of ethanol (CH₃CH₂OH)

Absorption

Area under the peaks tell us how many hydrogens we have.

TMS

Chemical shift δ/ppm

3   **Utilize a technique to analyze the surface morphology of the sample. Illustrate the principle and the working involved in it.**

OR

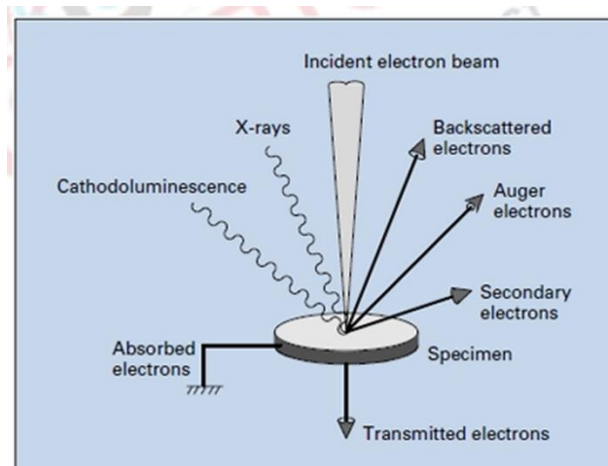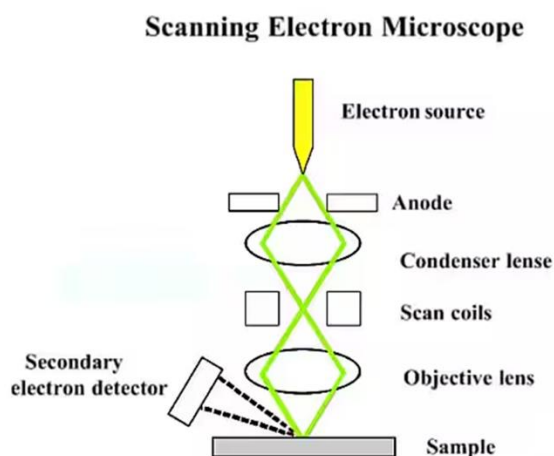**Describe the working principle of Scanning Electron Microscope with a block diagram.**

Scanning Electron Microscope a characterization tool used to find the surface morphology and atomic number.

Principle: When an electron beam strikes the sample surface produces back scattered electrons if the interactions are elastic and provide information of atomic number, higher the atomic number of an element brighter is the material image. Similarly, inelastic interactions produce Secondary electrons, which provide morphology of a sample.

Instrumentation:

Scanning Electron Microscope

Electron source

Anode

Condenser lense

Scan coils

Secondary electron detector

Objective lens

Sample

Incident electron beam

X-rays

Cathodoluminescence

Backscattered electrons

Auger electrons

Secondary electrons

Absorbed electrons

Specimen

Transmitted electrons

Working: Tungsten filament produces electron beam and attracted by the anode. These electron beams are narrow down by condenser lens and focus on to the sample through the objective lens. **Scanning coils**, are used to raster the beam onto the sample. After interaction between incident electron beam and sample the secondary electrons are allowed to pass through detector and image is displayed by output device

4   Describe the working principle and uses of X-ray diffraction with block diagram.

OR

Apply Bragg's law and illustrate the block diagram of an X-ray diffraction instrument.

XRD is a technique used to study the structure, composition and properties of **crystalline** materials. It is based on diffraction of x-rays by the atoms in crystal lattice.
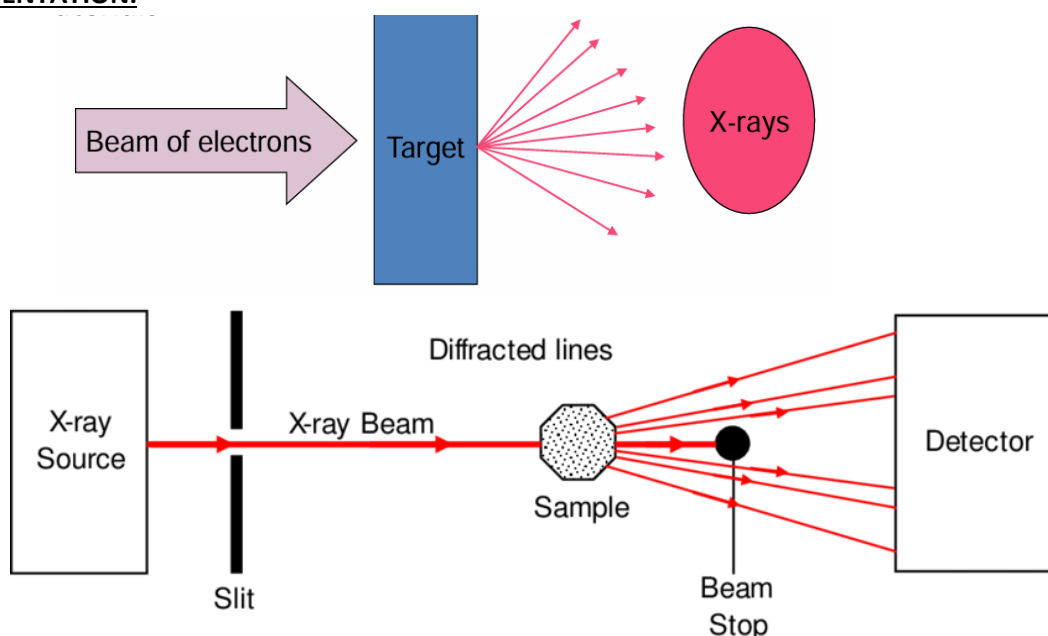
Principle: It works on the principle of Braggs Law which states that **when the x-ray is incident onto a crystal surface, its angle of incidence, θ , will reflect back with a same angle of** scattering, θ.

It is represented as nλ =2dsinθ
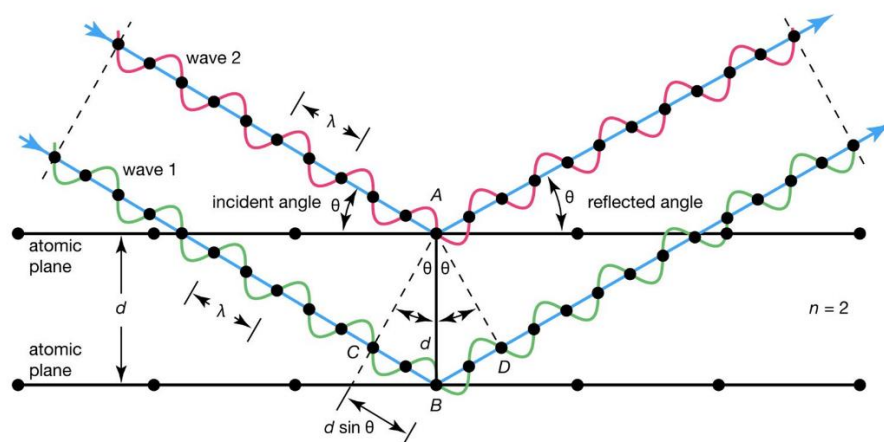
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INSTRUMENTATION:

Working:

The high intensity nearly monochromatic Kα x-rays (λ=1.54 A°)are directed at a crystal interacts with the electrons of the atoms in the crystal. The electrons oscillate under the influence of the incoming X-Rays and become secondary sources of EM radiation. The secondary radiation is in all directions. If the waves emitted by the electrons have the same frequency as the incoming X-rays- coherent. The emission can undergo constructive or destructive interference. Diffraction from different planes of atoms produces a diffraction pattern, which contains information about the atomic arrangement within the crystal.

APPLICATIONS :- structure, composition, purity of sample and phase analysis

5 Explain the principle, instrumentation, and working of conductometry in the estimation of a weak acid using a strong base as an example.

OR

Explain the conductometric estimation of a weak acid.

The electrochemical method of analysis used for the determination of the electrical conductance of an electrolyte solution by means of a conductometer.

Theory: Conductometry is based on Ohm's law. According to ohm's law the current flowing through the conductor is directly proportional to voltage and inversely proportional to the resistance.

$$E = I R$$

Specific conductance is also called conductivity. $\kappa = \dfrac{1}{A} \times C$

Principle: Conductomertic titrations are based on the measurement of conductance of solution, which is mainly depends on number of ions, charge on the ions and mobility of the ions. During the titration fast moving ions are replaced by slow moving ions and formation of ionic salt sodium acetate slightly increases the specific conductance. After neutralization point conductance increases due to hydroxyl ions.

$CH3COOH + NaOH \longrightarrow CH3COONa + H2O$
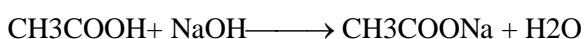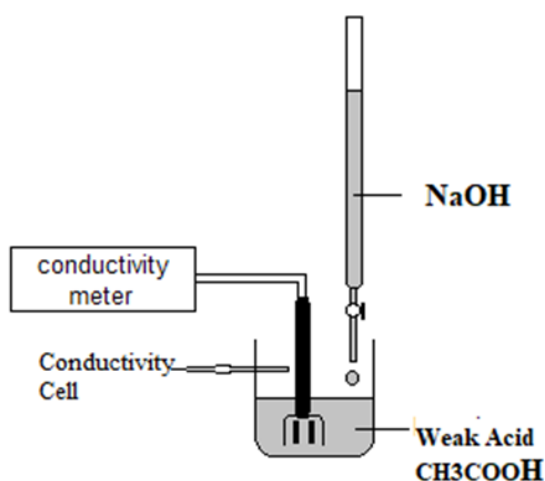
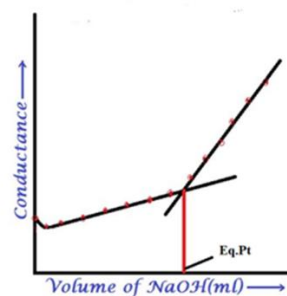| Instrumentation:	Procedure:
Fig:3.17 Instrumentation of Conductometry	Burette: Std NaOH (0.1N) Beaker: 50mL weak acid Device: Conductivity cell with Conductivity meter. Initial reading is taken with out the addition of NaOH and successive readings are taken for every 0.5ml addition of NaOH and 5 additional readings are taken after neutralization point. Volume of NaOH consumed was obtained from graph. Fig:3.18 Graph of weak acid Vs strong base

Tabular Column

Sl No	Volume of NaoH mL	Specific Conductance℧/cm

Calculation:

$N_{CH3COOH} = \dfrac{0.1 * Eq\ Pt}{50} = a$ Amount of CH3COOH = a * 60

6 **Discuss the principle, instrumentation and application of Potentiometry in the estimation of Iron.**

Potentiometric titrations involve the measurement of the potential of a suitable indicator electrode with respect to a reference electrode as a function of titrant volume.

Principle: Redox titrations can be carried out potentiometrically using platinum and calomel electrode combination in a manner similar to acid-base titrations.

For the reaction; $Reduced\ form \rightarrow Oxidised\ form + n\ electrons$

The potential is given by Nernst equation

$$E = E^\circ + \frac{0.0591}{n} \log \frac{[\text{Oxidised form}]}{[\text{Reduced form}]}$$

$$E = E^\circ + \frac{0.0591}{n} \log \frac{[Fe3+]}{[Fe2+]}$$
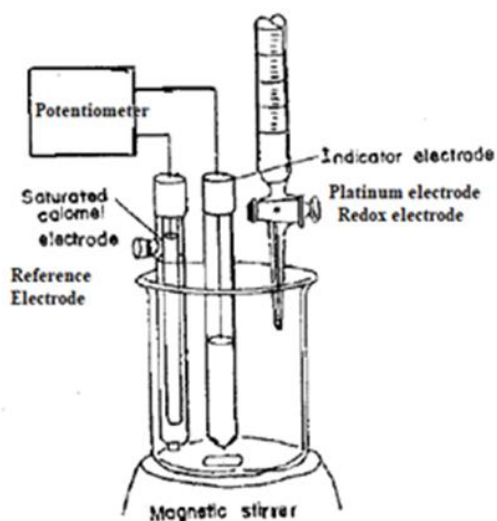
Instrumentation:

Fig: 3.19 Instrumentation of Potentiometry

Working

Platinum electrode: It is an indicator electrode, used to measure the potential of the analyte solution comparing with that of reference electrode.

Calomel electrode: It is a reference electrode and used for the determination of the analyte by maintaining the fixed potential.

Burette: Standard $K_2Cr_2O_7$ solution.

Beaker: Test solution (FAS) and 5ml of dilute Sulphuric acid($K_2Cr_2O_7$ is a strong oxidizing agent in acidic medium)

The electrodes are connected to potentiometer and initial potential was recorded without the addition of $K_2Cr_2O_7$. $K_2Cr_2O_7$ was added in the increments of 0.25 ml from the semi micro burette and corresponding emf was recorded. This process was continued till there was a sudden rise in the potential

and additional five more readings were recorded after sudden rise in potential. A graph was plotted by taking $\frac{\Delta E}{\Delta V}$ along y-axis and volume of $K_2Cr_2O_7$ along X-axis. Amount of FAS was calculated using equivalent point.

Vol. of $K_2Cr_2O_7$ (V) ml	Emf E (mv)	ΔV $(V_2 - V_1)$ ml	ΔE (E_2-E_1)	$\Delta E/\Delta V$ (mv/ml)
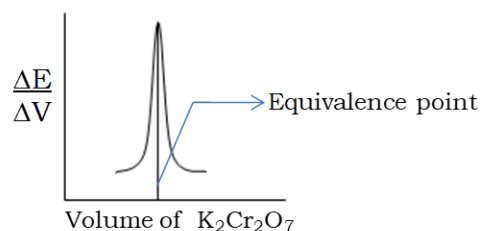
Fig:3. 20 Graph of potentiometry

Calculation: $N_{FAS} = \frac{(NV)_{K_2Cr_2O_7}}{V_{FAS}} = a$

Amount of FAS per liter $= N_{FAS} \times \text{Eq.Wt.of FAS (392)}$

392 g of FAS contains 55.85 g Fe

a g of FAS contains $\frac{55.85 \times a}{392} = \ldots\ldots\ldots\ldots g$ of Iron