LITTLE FLOWER COLLEGE
DEPARTMENT OF CHEMISTRY

TOPIC : X-RAY PHOTOELECTRON SPECTROSCOPY(XPS)

PRESENTED BY

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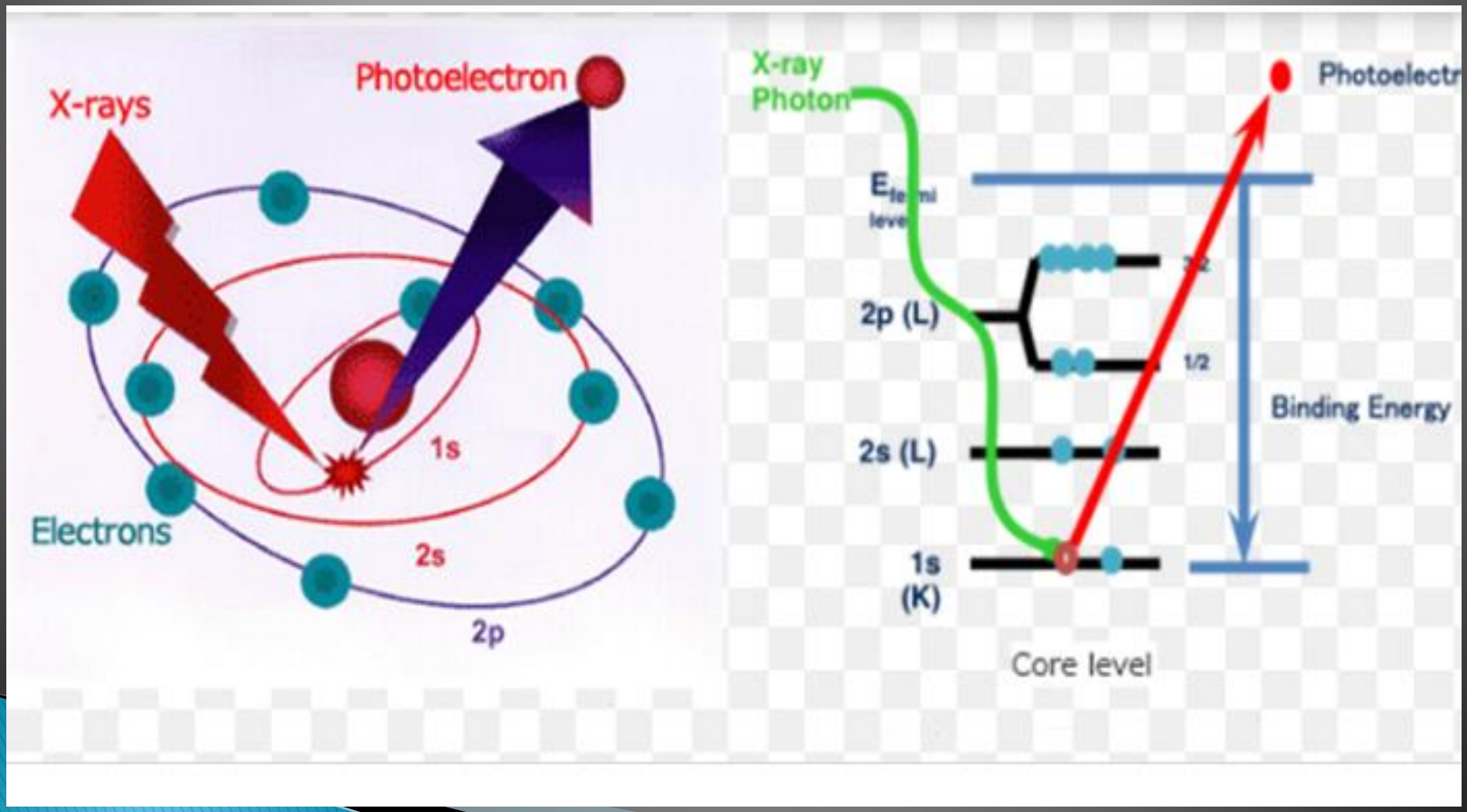
PRINCIPLES

- The technique is also called as ESCA .
- This technique not only provide information regarding the atomic composition of sample , but also information about structure and oxidation state of compounds.
- This is based on the photo electric effect.
- ESCA is used to determine chemical states of element including chemical behavior.
- It also reveals the chemical composition and chemical states of atom on surfaces of solid.

When atoms or molecules are subjected to high energy radiation, photons in the radiation collide with electron in the sample as a result in which electron is ejected.



The process is denoted as,

$$A + h\nu = A^{+*} + e^{-}$$

A is a atom ;molecule; or ion

A+ is the electronically excited ion with a positive charge one greater than that of A

The ejected electron depart having different velocities and the XPS measures the velocity distribution of released electron.

Each electron is held in place by nucleus with characteristic binding energy.

The typical value of B.E of electron in valence level of atoms lie in range of 5-30 eV

When the energy of photon imparted is greater than BE then the leaving electron will have certain kinetic energy and velocity.

$$h\nu = BE + KE$$

Electrons may be ejected from core or valence level of atom .

For ionization of core electron higher energy is needed . So X- ray source is used.

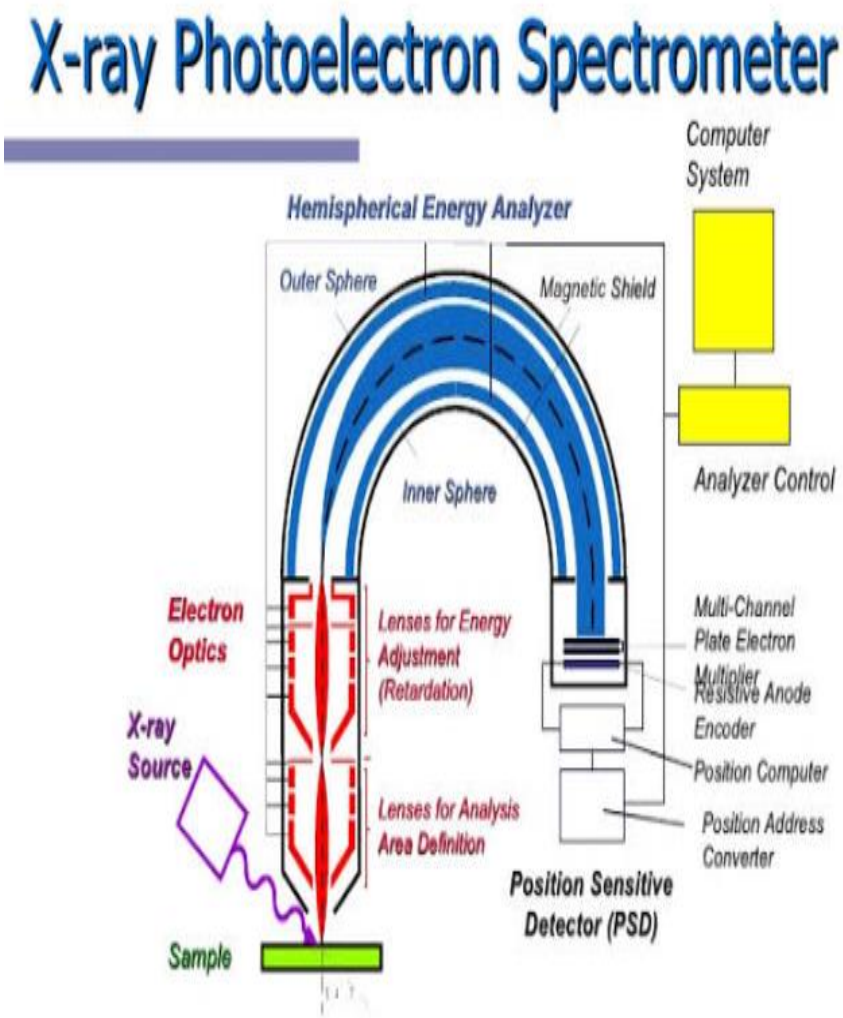
X-ray beam is produced by electron bombardment of a metal target such as Al and Mg.

Because the electrons are chemically active detection of electron is carried out in high vacuum.

INSTRUMENTATION

PARTS

- Source
- Sample holder
- Energy analyzers
- Electron beam excitation
- Detector
- High vacuum system



SOURCE

- Simplest X-ray source are X-ray tubes equipped with magnesium or aluminum targets and suitable filters.
- It is used without a monochromator because of high intensity and narrow wavelength bands of Kα line of these light intensity.
- It should be noted that narrow band are desirable because of enhanced resolution.
- More sophisticated XPS make use of crystal monochromator inorder to provide an X- ray beam having a band with of 0.3eV
- Monochromators are used to examine much smaller spots in the surface.

SAMPLE HOLDER

- Solid samples are mounted in a fixed position as close to photon or electron source and the entrance slit of spectrometer as possible.
- The sample compartment is evacuated to pressure of 10^-5 torr in order to prevent attenuation of electron beam
- Better vacuum are required inorder to prevent contamination of the surface of sample by oxygen , water ,or it react or absorbed on surface.
- High pressure leads to excessive attenuation of electron beam due to inelastic collision.
- If sample pressure is low it leads to weak signals.

ENERGY ANALYZER

- Energy analyzer between sample and detector require more efficiency than filters because electron beam are of less intense.
- Retarding field analyzer – geometric restriction are required to ensure that all electron are moving normal to grid.
- Magnetic deflection analyzer – are effective but less convenient to use than electrostatic analyzer.
- Most widely used analyzer are either cylindrical or spherical electrostatic field analyzer.
- Most of electron spectrometers are hemispherical so that electron travels in curved path.
- Electrons of various KE can be focused on detector.

ELECTRON BEAM EXCITATION

- It can be used with or without monochromator
- Electron from heated cathode is accelerated by an electric field are homogeneous but are spread because they are emitted from cathode with range of KE
- For greater homogeneity energy filters are used.

DETECTOR

- Electron multiplier is used as detector because of sensitivity and convenience
- Channel electron multiplier is a small curved glass tube doped with lead and vanadium
- It accepts electron at one end and emit more electron at other acting as current amplifier
- When potential is applied across the material , pulse are produced for each incident electron
- This pulse is counted and the resolution element of electron spectrum is monitored and data stored in computer for subsequent display.

HIGH VACUUM SYSTEM

- Vacuum system avoid collision between photoelectrons and gas molecules in the spectrometer and minimize surface contamination from residual gases
- Ultra high vacuum is characterized by pressure lower than about 100 nano scale.

ADVANTAGES

- Non – destructive technique
- Surface sensitive (10 – 100 angstrom)
- Quantitative measurements are obtained
- Provides information about chemical bonding
- Elemental mapping

DISADVANTAGES

- Very expensive technique
- High vacuum is required
- Slow processing (half to 8 hours per sample)
- Large area analysis is required
- H and He can not be detected
- Data collection is slow 5 to 10 min
- Poor lateral resolution

USES

- To measure elemental composition of the surface
- Chemical or electronic state of each element in the surface
- Uniformity of composition across the top of surface
- Uniformity of elemental composition as a function Of ion beam etching.

APPLICATION

- Polymer surface
- Corrosion
- Adhesion
- Semiconductors
- Dielectric materials
- Thin film coating

THANK YOU