

QUESTIONS FOR NPTEL: MODERN INSTRUMENTAL METHODS OF ANALYSIS BY  
Dr.J.R.MUDAKAVI

Lecture 1:

1. Explain the importance of analytical science in solving day to day problems.
2. Indicate the critical role of analytical instrumentation vis-à-vis (i) sports (ii) Oil Spills

Lecture 2:

1. Indicate the current knowledge of atomic nuclear structure along with the fundamental properties.
2. Compare Bohr's and Sommerfeld's atomic models.

Lecture 3:

1. What is the current state of knowledge with respect to electronic structure in the elements?
2. Explain how the modern periodic table is in consonance with atomic number and electronic structure.
3. Indicate various processes occurring when electromagnetic radiation interacts with matter.

Lecture 4:

1. Explain the concept of 'band pass width' in spectroscopic instruments.
2. Indicate diagrammatically the phenomenon of emission, absorbance, fluorescence.
3. In what way chemiluminescence differ from fluorescence?

Lecture 5:

1. Indicate various regions of electromagnetic radiation along with their energy levels and structural changes that occur upon irradiation of matter. Also indicate the corresponding spectroscopic techniques.
2. What type of molecular transitions can you expect when em radiation interacts with electrons in bonded and non bonded configurations in organic molecules?
3. Indicate Woodward – Feiser rules for aromatic compounds

### Lecture 6:

1. Calculate  $\lambda_{\max}$  for 2-hydroxy, 3-one- p-xylene using Woodward Feiser rules.
2. Calculate  $\lambda_{\max}$  for  $\delta$ -methoxy tetralone (76nm)
3. Calculate  $\lambda_{\max}$  for p-chloro acetophenone (254 nm)

### Lecture 7:

1. Derive Beer – Lambert's Law from the fundamental considerations.
2. Explain the concept of relative concentration error and its application in real analysis.
3. Explain the merits and demerits of deuterium, tungsten, tungsten iodide and mercury lamps as the light sources in UV-visible instrumentation.

### Lecture 8:

1. What are the various ways of securing a narrow wave length electromagnetic radiation in UV-visible range?
2. Bring out the special features of prism mountings in UV-visible instruments.
3. What do you understand by (i) Etalons (ii) Eschelle gratings and blazed holographic gratings?

### Lecture 9:

1. Explain the concept of signal modulation in spectroscopic instruments
2. Explain with the help of neat diagrams the working of divide array detectors
3. Explain the working of a photo multiplier tube

### Lecture 10:

1. How would you adopt a spectrophotometer for the color measurement of a painted surface?
2. How can you ascertain the composition of a complex using UV-visible spectroscopy?
3. What do you understand by flow injection analysis? How can you use it for solving production related problems?

### Lecture 11:

1. Explain the concept of quantum yield.
2. Indicate the conditions beneficial for fluorescence to occur in organic compounds.
3. In what way luminescence instruments differ from fluorescence instruments?

### Lecture 12:

1. Derive a relationship between fluorescence and concentration of a substance.
2. Draw a schematic diagram of a filter fluorimeter.

### Lecture 13:

1. Explain the constraints of using a xenon lamp as a radiation source in fluorescence instruments. What are its advantages?
2. Explain why fluorescence measurements of a compound are arbitrary?

### Lecture 14:

1. The first maxima for Bragg's diffraction from KCl crystal ( $d=0.314\text{nm}$ ) appears at  $14^\circ$ . Calculate the energy of incident x-rays (41.5 keV)
2. Calculate the distance 'd' in rock salt if its density is 2.18 g/cc and molecular weight is 58.5.
3. What do you understand by K series and L series of emissions in X-rays?
4. Explain the construction and working of X-ray detectors.

### Lecture 15:

1. Explain instrumentation diagram of XRF analytical technique.
2. What do you understand by single channel, double channel and multichannel EDXRF?

### Lecture 16:

1. What do you understand by Fraunhofer lines?
2. What do you understand by Einstein Emission, Coefficient?
3. Why line broadening occurs in AAs?
4. Draw an energy level diagram for sodium.

### Lecture 17:

1. Draw a schematic diagram of single beam, double beam AC, DC, AAS instruments.
2. What are the various mechanisms operative during line broadening?

### Lecture 18:

1. Explain the concept of LS, JJ Coupling in AAS?
2. Calculate the concentration of the excited cadmium ions at  $1000^\circ\text{C}$ ,  $1500^\circ\text{C}$  and  $3500^\circ\text{C}$ .
3. Why radiation sources are unique in AAS?

### Lecture 19:

1. Compare and contrast the premix burners and total burners in AAS. What are their advantages?
2. Describe the structure of a flame along with the chemical species being produced in a reducing flame?
3. Write a short note on atomization in AAS.

### Lecture 20:

1. Explain the various processes occurring during atomization along with the associated chemical reactions.
2. Explain the significance of signal to noise ratio in AAS measurement.

### Lecture 21:

1. Indicate various types of interferences occurring in AAS.
2. Why observation height is important in AAS?
3. What do you understand by the term (i) spectral interferences and (ii) volatilization interferences?

### Lecture 22

1. Explain the mechanism of non-spectral background correction using a schematic diagram.
2. What is a spectral interference? How can spectral interferences be eliminated using Zeeman Effect?
3. What are the different ways in which Zeeman Effect can be employed in AAS?
4. What do you understand by ionization interferences?

### Lecture 23

1. Explain how complexing agents can be employed to advantage in AAS?
2. What do you understand by analyte occlusion?

### Lecture 24

1. Explain the principles of hydride generation AAS. List all the elements that can be determined by HGAAS.

2. Elaborate on the effect of acids on the determination of hydride forming metals in AAS.
3. What are the essential changes in instrumentation to be made in hydride generation AAS?

#### Lecture 25

1. Explain the concept of cold vapour mercury analysis using the principles of atomic absorption.
2. Explain the similarities and dissimilarities of HGAAS and cold vapour mercury AAS.
3. Elaborate the instrumentation of cold vapour mercury AAS.

#### Lecture 26

1. Give an account of the evolution of graphite furnace atomic absorption spectrometry.
2. Why a high degree of atomization is required in GFAAS?
3. Give a schematic description of the LVOV's design and Mossman design in Electrothermal AAS.
4. Explain the concept of STPF in ETAAS.

#### Lecture 27

1. Indicate various phase reactions occurring in a graphite furnace during the electrothermal process
2. What do you understand by matrix modification?
3. What is vapour phase interference? How can it be avoided during ETAAS?

#### Lecture 28

1. Explain how plasmas can be generated.
2. Give a schematic diagram of Czerney-Turner, Ebert- Fastie, Rungekutta mountings in ICP- AES

#### Lecture 29

1. Explain how to introduce the sample in plasma AES?
2. What are the similarities and dissimilarities in ICP – AES and AFS.
3. What are the various types of interferences encountered in ICP – AES?
4. Bring out the differences between sequential and simultaneous ICP – AES analysis

### Lecture 30

1. Compare and contrast the advantages and disadvantages of Flame AAS, ETAAS and ICP-AES.
2. Indicate various models of vibrations a diatomic molecule exhibits.
3. The fundamental vibrational frequency of HCl is  $2890\text{ cm}^{-1}$ . Calculate the force constant of HCl. the atomic masses are  $1\text{H} = 1.673 \times 10^{-27}\text{ kg}$  ;  $35\text{ cl} = 58.06 \times 10^{-27}\text{ kg}$  (Ans =  $483\text{ Nm}^{-1}$ )

### Lecture 31

1. Explain the concept of Fourier Transform IR spectroscopy.
2. what do you understand by (i) apodization (ii) Fellgett advantage and (iii) multiplex advantage
3. What are the different ways of sample handling in IR?

### Lecture 32

1. Compare the merits and demerits of various sources of IR.
2. Give a schematic diagram of (i) Golay detectors, (ii) photon detectors and explain their working.
3. How can you differentiate between primary, secondary and tertiary amines using IR spectrum?

### Lecture 33

1. How can infrared spectroscopy be applied for refinery process industries?
2. Explain the mass spectrum of dodecane.
3. What are the basic chemical functions occurring in a mass spectrometer. Draw a schematic diagram of a mass spectrometer and explain the functions of each component.
4. Draw mass spectrum of isopropyl - n – pentyl- ether and write its various fragmentation modes.

### Lecture 34

1. Explain the origin of NMR.
2. Define chemical shift. How is it useful in NMR spectroscopy?
3. Explain various factors affecting the chemical shift?

#### Lecture 35

1. What do you understand by the term nuclear Overhauser effect? What is MRI?
2. Define (i) Galvanic cell (ii) Electrolytic cell (iii) liquid junction potential and (iv) Electrical double layer.

#### Lecture 36

1. Define electrodes of the first kind, second kind and third kind.
2. Indicate various electrochemical techniques employed for analytical purposes and the relevant electrochemical property being measured.

#### Lecture 37

1. Define concentration cell, Glass electrode and ion selective electrodes.
2. What type of chemical reactions is amenable for potentiometry?
3. Explain the functionality of quinhydrone electrode and antimony electrode.

#### Lecture 38

1. Draw a simplified circuit for potentiometric PH meter.
2. List the advantages and disadvantages of DME.
3. Plot the polarogram of cadmium in cadmium chloride
4. The diffusion current (i) for Zn is 4.125 when  $m = 16.25$  mg/sec and  $t = 4.3$  sec. If the diffusion current of an unknown solution is 4.3 mA calculate the concentration of zinc. ( Ans :  $4.02 \times 10^{-2}$  M)

#### Lecture 39

1. Explain the standard addition technique with reference to polarography.
2. Explain the use of KarlFischer reagent for moisture analysis in organic compounds.
3. What do you understand by dead stop titration?

#### Lecture 40

1. What are the various methods of chromatogram development.

2. Explain the concept of plate height in chromatography.
3. Define the terms resolution, partition coefficient, plate volume and retardation.

#### Lecture 41

1. Draw a schematic diagram of a gas chromatograph and explain the function of each component.
2. write short notes on : (a) columns and column materials (ii) flame ionization detector (iii) electron capture detector (iv) Programmed Temperature gas chromatography
3. Calculate the HETP if a column is 5-m long and average number of theoretical plates is 57. (Ans 2.8cm)

#### Lecture 42

1. Write short notes on thermal conductivity detector, and flame ionization detector.
2. Explain the concept and use of Kovat's index.
3. In what way the Reaction gas chromatography improves the efficiency of gas chromatographic analysis. Give at least five examples.

#### Lecture 43

1. What are the characteristic features of high pressure liquid compare Gas chromatography and HPLC as analytical tools.
2. Give a schematic diagram of HPLC – ms and GC-MS.
3. Write a short not on HPLC – MS & GC – MS.