

**VI SEMESTER**  
**ANALYTICAL INSTRUMENTS(EI 1352)**  
**Two marks Questions & Answers**

**UNIT I**

1. State Bouguer's law.

The mathematical statement which states that the radiant power absorbed is proportional to the thickness traversed is called the Lambert's law given by

$$\ln p_0 - \ln p = \ln(p_0/p) = k'b$$

2. State Beer's law.

The dependence of radiant power on the concentration of absorbing species can be given by Beer's law.

$$\ln p_0/p = k''c$$

Where  $p_0$  is the radiant power at  $c=0$  and

$p$  is the radiant power at  $c=c$  and

$c$  is the concentration of absorbing species

3. State Lambert-Beer law.

The relation representing

$\ln p_0/p = kbc$  Where  $p_0$  is the radiant power at  $c=0$  and

$p$  is the radiant power at  $c=c$  and

$c$  is the concentration of absorbing species

4. State the process involved in AAS.

The AAS phenomenon can be divided into two major processes

1. The production of free atoms from the sample
2. The absorption of radiation from an external source by these atoms

5. Name the types of detectors used for IR spectrometry.

1. Thermal detectors
2. Photon detectors

6. Define spectroscopy.

Spectroscopy is the measurement and interpretation of radiation emitted, scattered or absorbed by different atoms, molecules & other chemical species

7. Name the different types of spectrophotometers.

1. UV-visible spectrophotometers.
2. Infrared spectrophotometers.
3. FIFR spectrophotometers.

4. Atomic absorption spectrophotometers.
5. Flame emission spectrophotometers
  
8. What are the light sources used for AAS?
  1. Hollow cathode lamp
  2. Electrodeless discharge lamp
  
9. Give any two applications of flame emission spectrometry.
  1. FES is used in the determination of trace metals in liquid samples.
  2. FES finds wide application in agricultural and environmental analysis, industrial analysis of ferrous metal and alloy as well as glasses .
  
10. Specify the classification of IR region of spectrum.
  1. photographic region
  2. very near IR region (overtone region)
  3. near IR region (vibration rotation region)
  4. far IR region (rotation region)
  
11. Name the instruments used in IR spectrometry.
  1. IR radiation sources
  2. monochromators
  3. sample cells
  4. detectors.
  
12. Name few IR radiation sources.
  1. incandescent source
  2. nernst glower
  3. globar source
  4. mercury arc.
  
13. Give the advantages of grating monochromators
  1. gratings can be made with materials like aluminium which are not affected by moisture.
  2. grating monochromators can be used over wide wavelength ranges
  
14. Give 4 different techniques used for sampling of solids.
  1. solids run in solution
  2. solid film techniques
  3. null techniques
  4. pressed pellet technique
  
15. Name two different types of IR spectrometers
  1. dispersive IR spectrometers
  2. nondispersive IR spectrometers
  
16. Give the advantages and disadvantages of fourier transform IR spectrometers.

Adv:

1.FIFR methods are faster than dispersive instruments and hence specially useful in situations that require fast repetitive scanning.

2.FIFR provides increased energy throughput.

Disadv:

1.it is expensive than sequential dispersive instruments

2.for the precise movement of the mirror computer is also needed.

17. Specify the major design requirements of monochromators.

1.simplicity

2.resolution

3.spectral range

4.purity of exiting radiation

5.dispersion

18.Name the different mountings used in grating monochromators.

1. Littrow mounting

2. Ebert mounting

3. Czerny-Turner mounting

## Unit II

1. chromatography ,a short note.

Chromatography is defined as the physical and chemical method of separation between various components of a mixture into pure fractions or bands of each component.

2.what are the different types of gas chromatography?

The different types of gas chromatography are

➤ Gas liquid chromatography

➤ Gas solid chromatography.

3.Define retention time

The time required for each of the components to emerge from sample or mixture is called as the retention time.

4 Name the different parts of gas chromatography?

• Sample injection system

• Chromatographic column

- Thermostat
- Detector
- Recorder.

5. Explain the selection criteria for carrier gas.

The selection criteria for carrier gas are

- It should be very cheap
- It should have high thermal conductivity
- It should be inert
- The carrier gas should be selected according to the type of detector used.

6. Explain chromatographic column.

The column acts as the heart of a gas chromatography, where the fundamental process of separation takes place. Its action is based on the fact that when a sample of gas or vapour is introduced into the column, it spreads by molecular diffusion to give a concentration profile. As the sample moves through the column, additional spreading takes place. But, the band maintains the general shape, which is detected and recorded as a chromatographic peak.

7. What is pyrolysis?

Pyrolysis is an accepted method of handling solid samples. It extends gas chromatographic analysis to compounds such as rubber, soil, textiles, coals, resins, polymers, paint films etc.

The method lends itself to studies on heat stability and thermal decomposition. It is also called as controlled thermal fragmentation.

8. List some detectors in gas chromatography.

- Thermal conductivity detector
- Flame ionization detector
- Thermionic emission detector
- Electron capture detector
- Flame photometer detector
- Photo ionization detector
- Electrolytic conduction type of detection
- Dual detector

9. Give the principle of Gas-Solid chromatography.

When a gas or vapour comes in contact with an adsorbent, certain amount of it gets adsorbed on the solid surface. This takes place according to a phenomenon called Langmuir phenomenon given by

$$x/m = k_1c + k_2c^2$$

where  $k_1, k_2 \rightarrow$  constants

$x \rightarrow$  mass of gas or vapour adsorbed

$m \rightarrow$  mass of adsorbent

$c \rightarrow$  vapour concentration in gas phase

10. Give the principle of Gas-Liquid chromatography.

If the vapour or gas comes in contact with a liquid, a fixed amount of it gets dissolved in the liquid. This takes place according to Henry's law of partition given by

$$x/m = kc$$

where  $m \rightarrow$  mass of liquid used

$c \rightarrow$  vapour concentration in the gas phase

$x \rightarrow$  mass of gas

$k \rightarrow$  constant.

11. What are the advantages of gas chromatography?

The advantages of gas chromatography are

- Good accuracy and precision
- High sensitivity
- Apparatus cost is cheaper than liquid chromatography
- Shorter time of analysis
- Longer life of the instrument.

12. Write the features of thermal conductivity detector.

- ❖ It is simple, inexpensive, non-selective and non-destructive and displays a universal response
- ❖ Being non-destructive, the column effluent can be passed through a TCD and then into a second detector
- ❖ The sensitivity is 0.3 ng/ml. The linearity is between  $10^4$  to  $10^5$
- ❖ It is particularly suitable for fraction collection and preparative gas chromatography.

13. On what factor does the choice of detector will depend on liquid chromatography?

- Good sensitivity
- Better selectivity
- The detector must be able to operate in the presence of background signal
- The response time of the detector must be compatible with chromatography event.

14. What are the limitations of bulk property detector?

Limitations :

It is insensitive

It requires good temperature control.

15. Write the limitations of gas chromatography.

Only about 20% of known compounds lend themselves to analysis by gas chromatography. This is because the compounds are volatile and they cannot pass through the column. Also they are thermally unstable and decompose under certain conditions of separation.

16. What are the different types of liquid chromatography?

The different types of liquid chromatography are

- Liquid-liquid chromatography
- Liquid-solid chromatography
- Ion exchange chromatography or Bonded phase chromatography
- Exclusion chromatography

17. Name the different types of pumps used for mobile-phase delivery system in liquid chromatography.

The different types of pumps used for mobile-phase delivery system in liquid chromatography are

- Reciprocating pump
- Syringe type pump
- Constant-pressure pump.

18. What are the different types of columns used in liquid chromatography?

The different types of columns used in liquid chromatography are

- Standard column
- Radial compression column
- Narrow-bore column
- Short, fast column
- Guard columns and in-line filters.

### UNIT - III

1. What are the types of membrane sensor?

- Glass sensor
- Solid state sensor
- Solid matrix sensor

2. How measurements are done in ion selective electrodes?

Ion selective electrodes measure ion activities, i.e. measures the thermodynamically effective free ion concentration. In dilute solutions, ion activity usually approaches ion concentration.

3. Define conductivity of electrolyte?

The conductivity of electrolyte is a measure of the ability of the solution to carry electric current. The current through the solution takes place through the movement of electrically charged particles called ions.

4. Give the methods of measuring conductance?

- Null method
- Direct reading method.
- 

5. Why temperature compensation necessary in conductivity measurement?

The conductivity of electrolytic solution varies with temperature. This is because, the ionic mobilities are temperature dependent. The temperature coefficient is of the order of 1.5 to 2% at room temperature. So control of temperature is very essential.

6. Give the methods of measurements of Oxygen?

Methods used to measure Oxygen are classified as either Physical or Chemical methods. Physical method uses the paramagnetic property of Oxygen or thermal conductivity as the basis for quantitative determination. Chemical method includes potentiometric & catalytic combustion.

7. Give the working principle of electrical conductivity meter?

The instrument works on the following principle, that the conductivity of an aqueous solution depends on the inorganic impurity, & the instrument measures this in terms of resistance of a standard water column or tube through which the sample is passed. The conductivity is the reciprocal of resistance.

8. Give the application of Silica analyzer?

In thermal power plants, silica content is measured in steam before turbine. Silica analyzers are used for anion exchanger, effluent monitoring & effluents of mixed-bed exchangers.

9. What are the two measurements made in Silica analyzer?

- Chemical blank measurements
- Quantitative determination
- 

10. What is chemical blank measurement?

In this sequence the ammonia molybdate solution, sulphuric acid & reduction solution are simultaneously added to the mixing vessel. This solution is diluted with sample to a suitable volume & is then emptied to the measuring cuvette where it is measured & drained away. This is known as chemical blank measurement.

11. What is the use of blank in silica analyzer?

The reason for the use of blank on each cycle is to give the analyser long term stability by compensating for the effect of variables such as coloration of the sample or reagents, temperature, or aging of the lamp of photo cell.

12. Give the application of Sodium analyzer?

Sodium analysers find applications in thermal power plants for determining sodium ion concentration in boiler water, monitoring carry over detection of condenser leaks & the exhaustion of water treatment plant cation exchange units.

13. Why ammonia gas is added to the sample in Sodium analyser?  
The sodium selective glass electrode carries out the measurement in buffered solution above 10PH, where it responds specifically to value of the sample is sodium ion activity. So the PH must be adjusted to within desired range. The PH value of the sample is maintained in the flow cell by adding ammonia gas to the sample.
14. How PH of a solution is measured & give the Nernst equation?  
PH is measured by electro chemical cell, consist of two electrodes
- Measuring Electrode
  - Reference Electrode
- Measuring electrode is sensitive to hydrogen ions & Ref: Electrode is not sensitive to hydrogen ions. The PH is calculated by Nernst eqn: which is given as  $E = E^0 + \frac{2.303RT}{F}[P_{cH}]$ , where  $P_{cH}$  is the PH value deviation from 7.
- R is the gas constant
  - T is the absolute temperature
  - F is the Faradays constant.
15. Give the different types of electrodes used for PH measurements?
- Hydrogen electrode
  - Glass electrode
  - Calomel electrode
  - Combination electrode
16. Give the characterizes of glass electrode?
- Sensitivity is above 95%
  - Fast response
  - Low melting point
  - Relatively high electrical conductivity
  - High horoscopy.
17. Give the design criteria of PH meter?
- Input impedance of PH meter should be very high.
  - Current should not be drawn from the solution from the PH cell.
  - Temp: compensation must be provided with resistive thermo meter or by thermistor.
18. Give the general classification of PH cell?

- Null deflection type or potentiometric type.
- Direct reading type.

## UNIT IV

1. Define Thermal Conductivity of a gas?

Thermal Conductivity of a gas is defined as the quantity of heat (in calories) transferred in unit time (seconds) in a gas between two surface  $1 \text{ cm}^2$  in area when the temperature difference the surfaces is  $1^\circ\text{C}$ .

2. Why thermistors are used in thermal conductivity analyzer as a heat sensing elements?

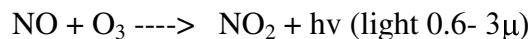
Thermistors possess the advantage of being extremely sensitive to relatively minute changes in temperature and have a high negative temperature coefficients. The speed of response is also high.

3. What are the applications of thermal conductivity gas analyzer?

- a. It is used in the measurement of hydrogen in blast furnace gases
- b. In the determination of argon in Oxygen in the process of air decomposition
- c. In the determination of sulphur dioxide in roasting gases in the production of sulphuric acid.

4) How is nitrogen-di-oxide prepared by chemiluminescence?

The nitric oxide reacts with ozone to form nitrogen-di-oxide with chemiluminescence .



5) What are the advantages of Hydrogen Sulfide analyzer?

- i) It do not require pumps or aspirators to pull in the sample
- ii) They are unaffected by wind or variations in relative humidity.

6) What is the use of gold films in  $\text{H}_2\text{S}$  analyzer?

Gold films absorb hydrogen sulfide and register the concentration by a proportional change in their resistance.

7) Where are the electrochemical sensors used?

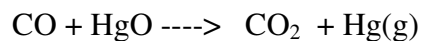
Electrochemical sensors are preferred in applications where the atmosphere is oxygen, where ruggedness is not important.

8. What is the principle of H<sub>2</sub>S analyzer?

Sample at a constant flow rate enters a humidifier where it bubbles through a 5% acetic acid solution. The sample then flows into the reaction window of the sample chamber, where it passes over an exposed surface of paper sensing tape impregnated with lead acetate. H<sub>2</sub>S reacts with lead acetate to form lead sulfide causing a brown stain on the paper. The rate of reaction and resulting rate of color change is proportional to the concentration of H<sub>2</sub>S in the sample.

9) What is the principle of CO analyzer?

It is based on the oxidation of carbon monoxide. It is oxidized by hot mercuric oxide.



The mercury vapor released may be measured photometrically.

10) What is the use of protective filter in Industrial analyzer?

In Industrial analyzer gas-handling components should always include a small protective filter, preceded by a major filter if the gas contains suspended matter that requires removal.

11) What is the use of stream drying equipment?

If a gas sample has a water vapour concentration high enough to cause condensation within the analyzer, or if moisture is an interferent, stream drying equipment is necessary.

12) What is the need of bypass pumping devices ?

The bypass pumping devices are needed to keep fresh sample rapidly supplied to the input of the analyzer.

13) What are the applications of oxygen analyzer?

- i) It is used in the areas of oxygen absorption studies on plants and tissues .
- ii) It is used in food processing industries.

iii) It is also used in respiratory studies.

14) What are the sources of error in oxygen analyzer?

- i) The filament temperature is affected by changes in the thermal conductivity of the carrier gas.
- ii) The cross tube must be horizontal to avoid an error due to gravitational chimney-flow effects.
- iii) Hydrocarbons and other combustible gases in the sample stream react on the heated filaments causes changes in temperature and therefore their resistance, which results in large error.

15) What is the principle of thermal conductivity analyzer?

It consists of a measuring cell and reference cell. When a gas whose thermal conductivity is lower is passed through the measuring cell, then the platinum wires in the measuring chamber are cooled less and their electrical resistance becomes more. The voltage across the diagonals is then proportional to the gas being measured.

16) Explain the different analysis methods of Nitrogen Oxide?

- i) Infrared
- ii) Ultraviolet
- iii) Chemiluminescent
- iv) Colorimetric
- v) Electrochemical

17) What are the applications of Electrochemical and Infrared sensors?

- i) Electrochemical sensors are used for ambient air monitoring
- ii) Infrared sensors are used for stack gas concentration

18) Explain the detection methods of Carbon Monoxide Analyzer?

- i) Non dispersive method
- ii) Mercury Vapour
- iii) Catalytic Oxidation
- iv) Electrochemical fuel cell

## UNIT V

1. What is the basic principle of NMR ?

Elementary particles such as electrons or nucleus behave as if rotatess about an axis and thus have the property of spin . The angular momentum associated with the spin of particle will be an integral or a half integral multiple of  $h / 2\pi$ , where h is Planck's constant.

2. Define NMR?

3. The study of absorption of radio frequency radiation by nuclei in a magnetic field ia called Nuclear Magnetic Rsonance(NMR).

The expression for the chemical shift is

$$\delta = \frac{H_{\text{sample}} - H_{\text{TMSi}}}{H_1} * 10^6$$

where  $H_{\text{sample}}$  and  $H_{\text{TMSi}}$  are the positions of the absorption peaks for the sample and reference mate.

3. Write any three applications of NMR Spectrometer.

I. Quantitative analysis :

II. Hydrogen Bonding:

**Elemental analysis:**

Here NBMR Spectrometer is used for determination of the total concentration of a given kind of magnetic nucleus in the sample.

4. Mention the limitations of NMR

1. Lack of Sensitivity.
2. While characterising the organic compounds, no information about the molecular weight is given, but the relative number of different protons present are only known.
3. In some compounds, two different types of H2 atoms at similar resonance frequencies resulting in an overlap of spectra .This makes difficult interpretation of such overlapped spectra.
4. In most of the cases,only liquids can be studied by NMR spectrometry.
5. What are the basic components of NMR Spectrometry?

The basic components of the NMR sptrometry are:

- A Magnet,produces magnetic field in the range 10,000 to 25,000 gauss
- Radio frequency transmitting system.
- The signal amplifier and detector.
- A display device, which may be a recorder or an oscilloscope.

- A non-magnetic sample holder ,which holds ths sample.

6. What is Mass Spectrometry?

Mass spectrometry is the most commonly used method which provides qualitative and quantative information about the atomic and molecular composition of organic and inorganic materials.It produces charged particles that consist of the parent ion and ionic fragments of the original molecule,and it sorts these ions acording to their mass/charge ratio.

7. Mention the advantages of Mass spectrometry.

- 1.Better Sensitivity.
2. More detailed understanding of kinetics and mechanisms of unimolecular decomposition of molecules.
3. Improved Speicificity in identifying unknowns or confirming the presence of suspected components.

8.What are the various parts of the Mass spectrometer?

- 1.Sample inlet system.
- 2.Ion source.
3. Ion acceleration and Mass analyser.
4. Ion collection system
- 5.Data handling system.
- 6.Vaccum system.

9. How can we obtain the NMR absorption spectra?

NMR absorption spectra can be obtained either by changing the frequency of the Radio frequency oscillator or by changing the spacings of the energy levels by varying the magntic field.

10.What is the principle behind the GM counter?

When an ionizing particle enters the counter, collision with the filling gas produces ion pairs. The formed ion pairs move towards the appropriate electrode under the voltage gradient. The mobility of electron is high and under this potential gradient it acquires sufficient velocity to produce new ion pairs by collision with atoms of argon. Repeating of this process produces an avalanche of electron moving towards anode. Electrons when striking generates ionization throughout the tube, called discharge. Each discharge builds a constant pulse counted by scale counter.

11. What is proportional counter?

When the electric field at the centre electrode of an ionization chamber is increased above the saturation level, but under that of the geiger region, the size of the output pulse from the chamber starts to increase but remains proportional to the initial ionization. A device operated in this principle is called a proportional counter.

12. What is depletion region?

A surface barrier detector consists of a p-n junction formed at the surface of a slice of a silicon. At the junction there is a planar region where there are no charge carriers and no electric field is called depletion region.

13. What are the two types of Semiconductor detectors?

- 1.Surface barrier Silicon detector.
- 2.Lithium drifted Silicon and Germanium detectors.

14. What is the dead time in GM counter ?

When the electron avalanche is collected on the anode, the positive ions, being much heavier, progress only a short distance on their way to cathode. Their time travel is about 200ns. During most of this time their presence as a virtual sheath around the anode efficiently lowers the potential gradient to a point where the counter is insensitive to the entry of more ionizing particle. This time is called dead time of GM counter.

16. Define Absorption, Fluorescence, Diffraction in X-ray Spectrometers?

Absorption of X-rays gives the information about the absorbing material just as in other regions of the spectrum.

Fluorescence emission of X-rays enables to identify and measure heavy elements in the presence of each other and in any matrix.

Diffraction of X-rays enables analysis of crystalline materials with a high degree of specificity and accuracy.

17. What are the basic instrumentation of X-ray spectrometer

- 1.X-ray generating equipment
- 2.Collimator
- 3.Monochromator
- 4.Detector

18. Why do we go for a solid state detector instead of scintillation detector, etc?

The electrical output from the solidstate is smaller hence a high gain, low noise amplification is required. To diminish noise pickup a preamplifier is generally located to the detector. Also scintillation counters suffer from poorer energy resolution. therefore, solid state detectors are mostly used.

## **ANALYTICAL INSTRUMENTS(EI 338)**

### **16 MARKS QUESTIONS**

#### **UNIT I**

- 1) Explain the single beam & double beam instruments used in UV spectrophotometer.

Ref : Instrumental methods of analysis  
Willard, Meritt, Dean, Settle

Page : 148 – 154

- 2) Draw & explain the schematic diagram of a typical double beam IR spectrometer.

Ref : Instrumental methods of analysis  
Willard, Meritt, Dean, Settle

Page : 300 – 301

- 3) What are non-dispersive spectrometers? Explain in detail the FTIR spectrometer. What are the advantages of the same?

Ref : Instrumental methods of analysis  
Willard, Meritt, Dean, Settle

Page : 301 – 305

- 4) Draw & explain the arrangement of the major parts of single beam & double beam Atomic absorption spectrometer.

Ref : Instrumental methods of analysis  
Willard, Meritt, Dean, Settle

## UNIT II

- 1) Draw the schematic diagram of a gas chromatograph & explain the components.

Ref : Instrumental methods of analysis  
Willard, Meritt, Dean, Settle

Page : 540 – 544

- 2) Discuss any two types of detectors used in Gas Chromatography.

- (i) Thermal conductivity detector
- (ii) Flame ionization detector

Ref : Instrumental methods of analysis  
Willard, Meritt, Dean, Settle

Page : 552 – 555

- 3) Explain in detail the following detectors used in Gas Chromatography.

- (i) Thermionic emission detector
- (ii) Electron capture detector

Ref : Instrumental methods of analysis  
Willard, Meritt, Dean, Settle

Page : 555 – 557

- 4) Draw & explain the instrumentation of HPLC in detail.

Ref : Instrumental methods of analysis  
Willard, Meritt, Dean, Settle

Page : 592 – 596

## UNIT III

- 1) With neat diagram, explain in detail the working principle of O<sub>2</sub>

Analyser based on magnetic susceptibility.

Ref : Handbook of Analytical Instruments  
-R.S. Khandpur

Page : 491 – 493

2) Draw & explain the schematic diagram of a NO-NO<sub>2</sub> analyser.

Ref : Process Measurements and Analysis  
-B.G.Liptak

3) With neat diagram, describe the working principle of a H<sub>2</sub>S analyser.

Ref : Process Measurements and Analysis  
-B.G.Liptak

4) Draw & explain the schematic representation of the method of measurement of dust concentration in stack.

Ref : Mechanical & Industrial Measurements  
-R.K.Jain

#### **UNIT IV**

1) Describe in detail about the constructional details and working principles of ion selective electrodes.

Ref : Handbook of Analytical Instruments  
-R.S. Khandpur

Page : 464 – 470

2) What are conductivity meters? Explain the principle of conductivity measurement by using the conductivity meters in detail.

Ref : Handbook of Analytical Instruments  
-R.S. Khandpur

Page : 427 – 431

3) Define pH. What are the design criteria of pH meters? Explain any one type of pH meter in detail.

Ref : Handbook of Analytical Instruments  
-R.S. Khandpur

Page : 448, 455 – 458

- 4) Explain with neat diagram a method of measuring oxygen dissolved in water.

Ref : Handbook of Analytical Instruments  
-R.S. Khandpur

Page : 464 – 470

## **UNIT V**

- 1) What is the basic principle of NMR? Discuss the working principle of NMR spectrophotometer & give its applications.

Ref : Instrumental methods of analysis  
Willard, Meritt, Dean, Settle

Page : 422, 431 – 437

- 2) Draw the block diagram of a pulsed Fourier Transform NMR spectrometer & explain its working principle.

Ref : Instrumental methods of analysis  
Willard, Meritt, Dean, Settle

Page : 437 – 439

- 3) Describe the working principle of GM counter & proportional counter in detail.

Ref : Instrumental methods of analysis  
Willard, Meritt, Dean, Settle

Page : 351 – 355

- 4) Draw the block diagram showing the major components of a Mass spectrometer & describe.

Ref : Instrumental methods of analysis  
Willard, Meritt, Dean, Settle

Page : 466 – 468