DEPARTMENT OF ELECTRONICS AND INSTRUMENTATION ENGINEERING
ANALYTICAL INSTRUMENTS
TWO MARKS QUESTIONS AND ANSWERS

UNIT- I

1. **Define Spectrophotometer?**
   Spectrophotometer are dispersive analyzers where a prism is used to separate the spectral components of the UV spectrum.

2. **State Beer-Lambert law.**
   The Beer-Lambert law is the fundamental law for quantitative analysis by absorption spectroscopy. This law states
   $$A = abc$$
   where A = absorbance, a = Molar absorptivity, b = Sample path length, c = Concentration of absorbing species.

3. **Name the main components of UV analyzer.**
   The main components of UV analyzer are Source, MonoChromator, Sample Cell, Detector, Readout Device.

4. **Give some example of radiation source used in UV region.**
   Cadmium, Mercury and Zinc Vapour sources that are used in the UV region are emission line sources.

5. **Name two sources used in NIR region.**
   i. Tungsten filaments ii. Quartzhalide lamps

6. **What is the use of O-rings?**
   Sealing of cell is accomplished with O-ring gaskets. Viton, Ethylene-propylene and Kalrez are commonly used.

7. **Name few detectors used in UV region.**
   i. Silicon photo diode ii. Photo Diode array

8. **Define path length.**
   The distance between the windows in the sample cell defines the path length.

9. **What is the purpose of sample cell?**
   The purpose of sample cell is to contain a representative sample from the process stream.

10. **Write the principle used in scanning spectrophotometer.**
    Scanning spectrophotometer are dispersive devices that normally utilize diffraction gratings to scan across a spectral regions. Scanning devices can be used for multiple component applications.

11. **Give some applications of Photo diode array.**
    i. Analysis of Hydrogen sulphide and sulphur dioxide in sulphur recovery
    ii. Determination of octane number.

12. **Give some applications of scanning spectrophotometer.**
    i. Measurement of multi component solvent mixture
    ii. Determination of octane number for gasoline samples.

13. **Write the applications of atomic absorption Spectrophotometry.**
    Mercury is conveniently analyzed by atomic absorption technique because the elemental vapour exists in atomic form under normal ambient conditions.

14. Define FTIR spectrophotometer?

FTIR spectrophotometer are dispersive devices that are being used for on-line analysis in the near infrared region. Instead of separating different wavelengths for measurement, the complete spectrum is encoded as an interferogram in a few seconds of measurement time, and the spectrum computed by fast Fourier transform.

15. Mention the application of Colorimeter.

Essentially, a colorimeter is a scientific instrument that measures the amount of light passing through a solution relative to the amount that passes through a sample of pure solvent.
Colorimeters have many applications in the fields of biology and chemistry.

16. Mention the application of flame photometry.

1) Analyse wide variety of materials such as environmental biological fluids, solids, plant materials and industrial cement, ferrous metals, glasses etc.
2) It is used for determining sodium, potassium, Aluminium, Calcium, and Iron in soil.

UNIT- II

1. What is Chromatography?

Chromatography describes a physical method of separation that is based on the difference in solubility (or adsorption) of substance between a mobile and stationary phase.

2. Define Chromatogram.

The individual components register a series of signals which appear as successive peaks above a base line on the recorded curve is called Chromatogram.

3. Name the detectors used in liquid Chromatography.

i. UV-Visible Spectrophotometric detectors ii. Fluorescence Detector iii. Refractive Index detectors iv. Adsorption Detector v. Electrical Conductivity detector vi. Thermal Detector

4. Name some source carrier gas used in Gas Chromatography.

Hydrogen, Helium, Nitrogen, Argon and CO2.

5. Write the limitations in FID.

i. Does not respond to inert gas and inorganic compounds
ii. The emerging compound get destroyed in the flame
iii. The response to sample weight has to be separately determined for each component.

6. Define retention time.

Time required by the component in the sample to emerge is called retention time.

7. Write the application of argon Ionisation detector.

Responds to most of the organic and Inorganic Compounds

8. Write the methods used in sample injection system in liquid Chromatography.

i. Syringe injection method ii. Injection valve method.

9. State the principle used in refractive Index detectors.

Refractive Index detectors depends on Snell's law. It is based on refraction, reflection or interference of light beams.

10. What is Pyrolysis?

Pyrolysis offers a technique for injection of certain types of materials which are low or non-volatile. This is a valuable technique for sample injection in rubber, plastics, polymers and adhesive industries.

11. Write the factors affecting Gas flow rate.

The rate of the gas flow depends upon column diameter. The flow rate is generally in the range 10-400ml/ minute .Very low and very high flow rate affect the efficiency adversely.

12. Define dead time and dead volume.

Dead time is the time required for a molecule of mobile phase to pass through the column.
Dead volume Vm is the volume of the mobile phase from the point of injection through the column to the detector.

13. Define thin layer chromatography.

A method of separating two or more chemical compounds in a solution through their differential migrations across a thin layer of adsorbent spread over a glass or plastic plate.

14. What are the application of HPLC?

1. It is applied in the process of isolation and purification of compounds. The information that is obtained from this includes identification, quantification and resolution of a compound.
2. Chemical separation can be accomplished using HPLC by utilizing the fact that certain compounds have different migration rates with respect to the particular column and mobile phase. The degree of separation is mostly determined by the choice of stationary and mobile phase.

15. What are the application of gas chromatography?

Gas chromatography is applicable for the analysis of the solute in both qualitatively and quantitatively.

16. What do you mean by open tubular column?

Capillary column are the open tubular column constructed from fused silica (a very high purity glass).The length of the column from 30 to 300m and a diameter of 1mm or less.

17. What is meant by liquid chromatograph?

Liquid chromatography is a technique used to separate a sample into its individual parts. This separation occurs based on the interactions of the sample with the mobile and stationary phases.

18. Write advantages of HPLC over gas chromatography.

i) Gas chromatography is in need of conversion of non volatile substances into volatile, such conversion is not needed in HPLC.
ii)Gas chromatography is not applicable to thermally unstable substances. but liquid chromatography can be applied to thermally unstable substances.

19. Define thin layer chromatography

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21. What are the properties of carrier gas?

1.The carrier gas must be chemically inert. That is otherwise it should not react with the sample or stationary phase. 2. It should be suitable for the detector employed and the type of sample analyzed. 3. It should give best column performance consistent with required speed of the analysis. 4. It should be free from the risk of fire.

22. Write the principle of adsorption detector.

This detector depends on the measurement of the evolution of the heat of adsorption and heat uptake at desorption as the solutes in the effluent stream comes in contact with an adsorbent.

23. Write the factors affecting Gas flow rate.

The rate of the gas flow depends upon column diameter. The flow rate is generally in the range 10-400ml/ minute .Very low and very high flow rate affect the efficiency adversely.

UNIT III

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 cm2 in area when the temperature difference the surfaces is 1°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?**
   i. It is used in the measurement of hydrogen in blast furnace gases
   ii. In the determination of argon in Oxygen in the process of air decomposition
   iii. 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 .
   $NO + O3 \longrightarrow NO2 + hv$ (light 0.6- 3u)

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 H2S 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 CO analyzer?**
   It is based on the oxidation of carbon monoxide. It is oxidized by hot mercuric oxide.
   $CO + HgO \longrightarrow CO2 + Hg(g)$
   The mercury vapor released may be measured photometrically.

9. **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.

10. **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.

11. **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.

12. **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.

13. 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,

14. 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.

15. What are the different analysis methods of Nitrogen Oxide?

i. Infrared
ii. Ultraviolet
iii. Chemiluminescent
iv. Colorimetric
v. Electrochemical

16. 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.

17. What is the principle of paramagnetic oxygen analyzer used for gaseous samples?

Paramagnetic refers attraction towards the magnetic field.Oxygen has the characteristics of attraction towards the magnetic field.Paramagnetic oxygen analyzer uses the magnetic properties of oxygen along with thermal conductivity for the measurement of oxygen in a gas.

UNIT IV

1. How measurements are done in ion selective electrodes?

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

2. 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.

3. 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.

4. Give the working principle of electrical conductivity meter.

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

5. 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.

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

- Chemical blank measurements
- Quantitative determination

7. Define 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 curvette where it is measured & drained away. This is known as chemical blank measurement.

8. 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.

9. 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 leakes & the exhaustion of water treatment plant cation exchange units.

10. Give the different types of electrodes used for PH measurements.

- Hydrogen electrode
- Glass electrode
- Calomel electrode
- Combination electrode

11. Define Ph.

Ph is defined as the negative logarithm of the ion concentration expressed in molarity.It is the activity of the hydrogen ion that is formally consistent with the thermodynamics of the Ph electromotive cell.

12. List the demerits of glass electrode.

i.It gives results for lower Ph values only.For higher Ph values the glass becomes responsible to sodium as well as to other cations.

ii.The electrode should be thoroughly washed with distilled water and should be rimsed with th test solution before taking the reading.

13. Give any two industrial application of ion selective electrodes.

i.Calcium ion electrode is used to find nerve conduction,bone formation,miscle contraction and in the cardiac contraction.

ii.Fluoride electrode is used in the determination of fluoride in bone,air,water and tooth pastes.

14. What is the electrochemical principle of blood glucose analyzer?

The glucose sensor works on the principle that the oxygen is consumed in the presence of glucose, providing a change in the signal from the conventional oxygen electrode. The glucose sensor is immobilized enzyme between the inner and outer membrane electrode. The sensor responds to the presence of glucose by changing the value of oxygen. The measure of change in oxygen value is a measure of glucose value.

15. Define bio sensor

A biosensor is a device that uses biological materials to monitor the presence of various chemicals in a substance or it can be defined as it is a device for the detection of an analyze that combines a biological component with a physiochemical detector component.

UNIT - V

1. What is Nuclear magnetic resonance?

The nuclei of atoms in a molecule on absorbing RF radiation may change their direction of spin.The analytical field involved with the interaction between the nuclei and rf radiation is called Nuclear magnetic resonance.

2. Define state of resonance.

When a nucleus is placed in a system where it absorbs energy, it becomes excited. It then loses energy to return to the unexcited state. It absorbs energy and again enters an excited state. This nucleus which alternately becomes excited and unexcited is said to be in a state of resonance.

3. Define Chemical shift.

The shift in the positions of NMR signals (compared with a standard reference) resulting from the shielding and deshielding by electrons are referred to as Chemical shift.

4. Write a method for measuring the Chemical shift.

To measure the magnitudes of chemical shifts of different kinds of protons, there must be some standard signal with respect to which the measurement can be made.For this purpose ,Tetramethylsilane(TMS),(CH3)4Si is used as the reference or standard compound.It gives only one signal which serves as fixed reference.

5. What are the factors that influence chemical shift?

The main factors which influence the chemical shift are ;

i. Inductive effect ii. Anisotropic effect iii. Hydrogen bonding

6. What is meant by Spin-Spin coupling?

The interaction between the spins of the neighboring nuclei in a molecule may cause the splitting of the lines in the NMR spectrum. This is known as spin-spin coupling which occurs through bonds by means of a slight unpairing of the bonding electrons.

7. Give some applications of NMR spectroscopy.

i.Structural Diagnosis

ii.Quantitaive analysis – used to determine the molar ratio of the components in a mixture.

iii Hydrogen Bonding – Used to study the hydrogen bonding in metal chelates as well as in organic compounds

iv.Structural determination

v.Intra molecular conversion

vi.Keto-enol Tautomerism

8. Write the limitation of NMR spectroscopy.

The limitations of NMR spectroscopy are :

i. Lack of sensitivity. The minimum sample size is about 0.1 ml having minimum concentration of about 1%

ii. In some compounds, two different types of hydrogen atoms resonate at similar resonance frequencies. This results in an overlap of spectra and makes such spectra difficult to interpret.

iii.Only liquids can be studied by NMR spectroscopy, although polymers, when preheated with various solvents, frequently become fluids which can be treated as liquids.

9. Define mass spectrum.

The compound under investigation is bombarded with a beam of electrons which produce an ionic molecule or ionic fragments of the original species. The resulting assortment of charged particles is then separated according to their masses. The spectrum produced is known as mass spectrum.

10. Define time of flight.

All ions leave the acceleration field with different velocities depending on their masses. With magnetic focusing, the ions gets separated by changing their directions. However, if these ions are allowed to travel in a straight line through a magnetic field – free region, they will take different times to travel a given distance. The measurement of "this time of flight" FORM the basis for the nonmagnetic separator.

11. What are the different types of mass spectrometer?

i. Magnetic Deflection mass spectrometer ii. Time of flight mass spectrometer iii. Double focusing mass spectrometer iv. Quadrupole mass spectrometer.

12. What are the different components of a mass spectrometer?

i. Inlet Sample System ii. Ion Sources iii. Electrostatic Accelerating System iv. Ion detectors and Recording of mass spectrograph v.Vaccum System

13. Write the principle of Electron Spin Resonance (ESR) spectrometer.

Electron Spin Resonance involves detecting the detection of a physical phenomenon of absorption of electromagnetic radiation in the microwave region by paramagnetic species that are subjected to magnetic field.

14. Write the components of electron spectroscopy.

i. A source of radiation to excite the sample. ii. An electron energy analyzer. iii. An electron detector.
iv. Read out system. v. High vaccum system.

15. Give some applications of mass spectrometry.

i. Molecular mass can be accurately determined by the mass spectrometry.
ii. It is used to determine the amount of component of a complicated mixture.
iii. Method for the detection of impurities.
iv. Polymers can be characterized by mass spectrometry.
v. Molecular formulae can be determined from the mass spectrum either partially or exactly.

16. What are the types of NMR spectrometer.

i. Minimal type NMR spectrometer ii. Multipurpose NMR spectrometer iii. Wideline NMR spectrometer

17. Write the advantages and disadvantages of time of flight mass spectrometer.

Advantages – It is simple and ruggedness.
It can handle unlimited mass range.
Disadvantage – Limited resolution and sensitivity.

18. What are the types of detectors in mass spectrometer?

i. Electron multiplier ii. Faraday cup collector iii.Photographic plates

19. What are the two common designs of Double focusing mass spectrometer?

i. Nier - Johnson design ii Mattauch - Herzog geometry

PART – B

UNIT – I

State and derive Beer's law from basic principles. Discuss the limitations of it.
2. Explain single and double beam instruments used in UV spectrophotometers.
3. Explain single beam and double beam photometer.
4. With a schematic diagram explain the atomic absorption spectroscopy.
5. Explain flame emission photometer with its instrumentation.
6. Explain the properties of electromagnetic radiation.
7. Explain monochromators and detectors.
8. Explain about photo multiplier tube.
9. Explain Fourier transform infra-red spectrometers.

UNIT – II

1. Discuss the principle used in gas-liquid chromatography and the process chromatography.
2. Describe the operation of a Gas – liquid, chromatography with a suitable diagram and discuss its applications.
   Draw the schematic diagram of a gas chromatography and explain the different parts in Gas – chromatography.
3. Describe a gas liquid chromatography with a schematic diagram. Explain how it works.
4. Give a typical chromatogram. Discuss some applications of gas liquid chromatography.
5. Explain in detail the following detectors used in Gas Chromatography.
   a).Thermionic emission detector b).Electron capture detector
7. With a neat schematic diagram discuss the separation principle of HPLC .
8. What are the requirements of HPLC pumping system and enumerate the application of HPLC.

UNIT – III

1. With a suitable diagram Explain the construction and working principle of oxygen analyzer .
2. Explain how the CO analyzer is used.
3. Explain how the NO2 analyzer is used.
4. Explain how the H2S analyzer is used.
5. Explain how the dust and smoke analyzer is used.
6. Explain how the IR analyzer is used.
7. With a suitable diagram Explain the construction and working principle of thermal conductivity analyzer.
8. What are the sources of air pollution and explain in detail?
9. What are the type's gas analyzers? Explain anyone with example.

UNIT – IV

1. Describe how a conductivity cell is used. Give the applications of conductivity measurement of a liquid.
2. Explain how the oxygen analyzer is used.
3. Describe the construction of a pH electrode. Draw the electronic circuit diagram for measuring pH of a liquid and explain its working.
4. Discuss how pH values are measured. Explain the role of calomel electrodes in this measurement.
5.Briefly explain about dissolved oxygen analyser.
6.Discuss in detail the principle, characteristics of electrodes used in pH meters.
7.Describe with a neat sketch, the principle of operation of water purity meter.
8.Discuss the method of measuring electrical conductivity of a liquid. Also describe the techniques for determining the purity of water.

UNIT – V

1.Draw the block diagram of an NMR spectrometer. Explain the function of each part and explain how it is used to obtain NMR spectra. How are these spectra useful?
2.What are mass spectrometers? Describe magnetic deflection mass spectrometers.
3.Explain time of flight and quadrupole mass spectrometers.
4.Describe the working of double beam mass spectrometer and give its applications.
5.Explain the instrumentation and applications of Scanning Electron Microscope (SEM) and Transmission Electron Microscope.
6.Describe how various samples are analyzed using NMR spectrometer with neat diagram.
7.Explain the construction and working principle of Electron Spin Resonance (ESR) spectrometer with neat diagram.
8.Explain the construction and working of Radio frequency spectrometer with neat diagram.