



Important Instructions to examiners:

- 1) The answers should be examined by keywords and not as word-to-word as given in the model answer scheme.
- 2) The model answer and the answer written by candidate may vary but the examiner may try to assess the understanding level of the candidate.
- 3) The language errors such as grammatical, spelling errors should not be given more Importance. (Not applicable for subject English and Communication Skills.)
- 4) While assessing figures, examiner may give credit for principal components indicated in the figure. The figures drawn by candidate and model answer may vary. The examiner may give credit for any equivalent figure drawn.
- 5) Credits may be given step wise for numerical problems. In some cases, the assumed constant values may vary and there may be some difference in the candidate's answers and model answer.
- 6) In case of some questions credit may be given by judgments on part of examiner of relevant answer based on candidate's understanding.
- 7) For programming language papers, credit may be given to any other program based on equivalent concept.

Q. No.	Question & Model Answer	Remark	Total Marks
1. a).	Attempt (any 3)		12
i)	State the use of buffer solution in blood pH measurement		4
Ans:	<p>1. Buffer is a substance which by its presence in a solution is capable of counteracting pH changes in the solution as caused by the addition or removal of hydrogen ions.</p> <p>2. Buffer solutions are characterized by their pH value.</p> <p>3. Buffer solutions used in blood pH measurement: 0.025 molar potassium dihydrogen phosphate with 0.025 molar disodium hydrogen phosphate and 0.01 molar potassium dihydrogen phosphate with 0.04 molar disodium hydrogen phosphate.</p> <p>These are used:</p> <ol style="list-style-type: none">1. To create and maintain desired stabilized pH in a solution2. To standardize the electrode chains for pH measurements.	4 marks for any relevant 4 uses	



ii)	Explain the elements of analytical instruments with the help of block diagram.		4
Ans:	<pre>graph LR; A[Chemical information source] --> B[transducer]; B --> C[Signal conditioner]; C --> D[Display system]</pre> <ul style="list-style-type: none">• Chemical information source generates a set of signals containing necessary information• Transducer converts information from the chemical source to electrical quantities• Signal conditioner converts the output of the transducer into an electrical quantity suitable for transmission or display. It modifies or conditions the signals so that the signals will be suitable for further transmission or processing.• Display system provides a visual representation of the measured quantity as a display on a chart or CRT or recorder.	BD 2 mark, explanation 2 marks	
iii)	Give any 4 applications of liquid chromatography.		4
Ans:	<ul style="list-style-type: none">• Biochemical Screening for Genetic Disorders,• Analysis of biological fluids,• Therapeutic Drug Monitoring and Toxicology,• R&D in pharmaceutical industries,• Vitamins and Related Metabolites,• Steroid Hormones	(any 4, 1 mark each)	
iv)	State 4 major gas pollutants along with their typical concentration.		4
Ans:	Major gas pollutants are carbon monoxide, sulphur oxide, hydrocarbons, nitrogen oxides, oxidant etc. Carbon monoxide: Its average concentration is below 200 ppm. Hydrocarbon: the various sources of hydrocarbon are	any 4,1 mark each	

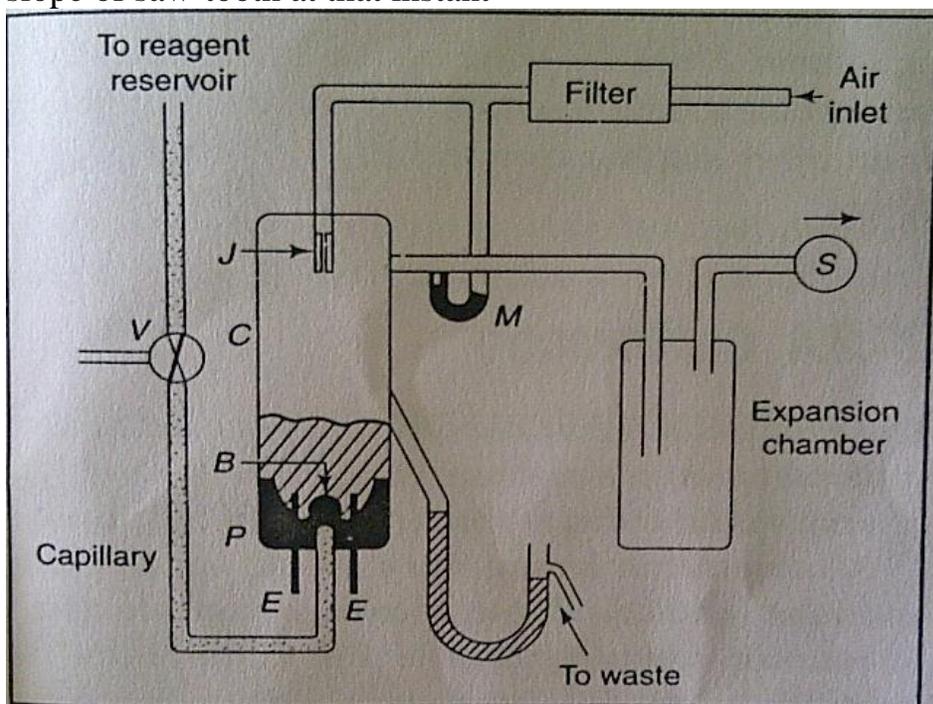


	<p>petroleum-refining process, incomplete combustion and evaporation of fuels. Methane is the major component of total hydrocarbon emission.</p> <p>Oxidants: the major component is ozone, which has damaging effect on plants and animals</p> <p>Sulphur dioxide: Its concentration in urban areas is 0.024 ppm.</p> <p>Nitrogen oxides: Its level ranges from 0.5 to 0.12 ppm.</p>		
b)	Attempt any one.		06
	i) What is monochromator? Explain working of prism as monochromator with the help of diagram.		
Ans:	<p>Monochromator is the wavelength selector in spectrophotometer. Spectrophotometer is used to measure the amount of light that a sample absorbs. It isolates monochromatic radiation in a more efficient manner with Monochromator. Thus the monochromatic light is obtained by allowing the light beam to pass through a Monochromator which are prism or diffraction grating. Thus the monochromator is the wave length selector.</p> <p>Parts: light source, prism monochromator, cuvette with sample, photocell detector.</p> <p>The light source is a 6V tungsten lamp which emits radiation in the wavelength region of visible light. Light is made to fall on the prism. The monochromatic light is obtained by allowing the light beam to pass through a prism monochromator. Prism is an optical component to disperse the light or modify the direction of light.</p> <p>The wavelength selection is done by rotating the prism about a pivot. The prism has an aluminized rear surface. Due to the prism, shorter wavelength is dispersed more.</p> <p>Light, after getting dispersed by prism, passes through the sample and then detected by the photocell.</p>	<p>Monochromator :explanation 2 marks,</p> <p>working of prism monochromator 2 marks,</p>	

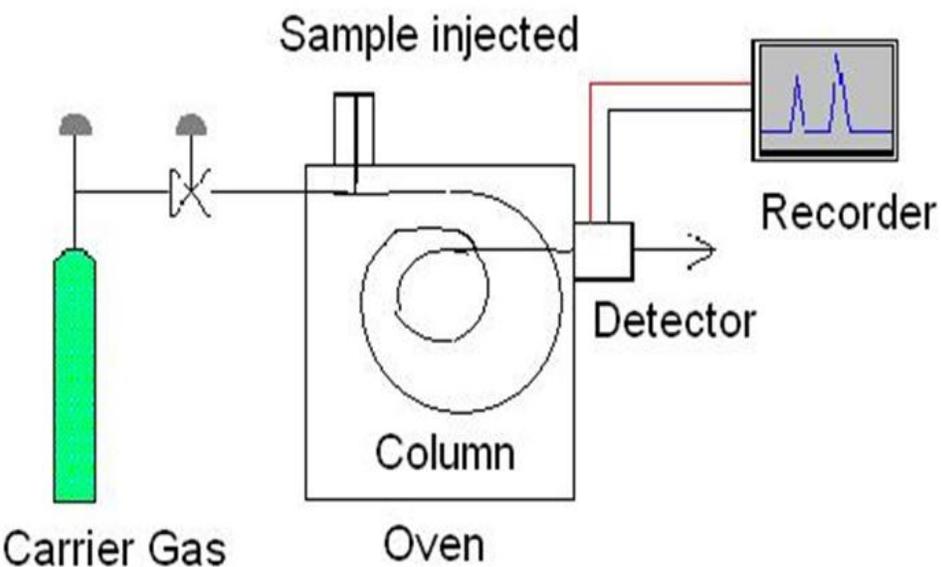


		<p>diagram 2 marks</p>	
<p>ii)</p>	<p>Describe the conductivity method for measurement of SO₂ in air with a neat labeled diagram.</p>		
<p>Ans:</p>	<p>When air sample containing SO₂ (sulphur dioxide) is passed through a solution consisting of sulphuric acid and hydrogen peroxide, its electrical conductivity changes due to formation of sulphuric acid by oxidation of SO₂.</p> $\text{H}_2\text{O}_2 + \text{SO}_2 \longrightarrow \text{H}_2\text{SO}_4 \longrightarrow \text{H}^+ + (\text{HSO}_4)^-$ <p>Adv: fast response & high sensitivity Disadv: interference by non-SO₂ gases which remove or produce ions in the solution affect the performance.</p> <p>Conductivity cell is used for continuous measurement of SO₂ in the air. It is made of glass, consists of a jet J, and orifice near the jet. It consists of 2 electrodes E, made of stainless steel wire. It is inserted through a cap P. The cap is sealed to the base of the cell. Reagent enters the cell from a central feed tube inserted in the cap. A small glass bead B in the cell acts as a non return valve on the entry of the central feed tube and prevents sulphuric acid diffusing from the</p>	<p>Explanation 3 marks,</p> <p>diagram 3 marks</p>	

cell. The end of the jet is made from a piece of capillary tube. A filter is placed before the jet to prevent blocking due to solid material. Since the cell is of small size, its capacity to absorb SO_2 is limited. Therefore electrolyte is discharged and replaced at regular intervals. 5 V AC is applied to the cell to measure the conductivity. AC avoids polarization. The resulting output current is recorded as saw-tooth waveform. Concentration of SO_2 at any instant is proportional to slope of saw-tooth at that instant



2.	Attempt (any 4)		16
a.)	Give the classification of chromatography. Explain any one of them in brief.		4
Ans:	Gas chromatography 2. Liquid chromatography a. Paper chromatography b. Column chromatography c. Thin layer chromatography	1 marks for classification	

	<p>Gas chromatography: Gas chromatography is a term used to describe the group of analytical separation techniques used to analyze volatile substances in the gas phase. In gas chromatography, the components of a sample are dissolved in a solvent and vaporized in order to separate the analytes by distributing the sample between two phases: a stationary phase and a mobile phase. The mobile phase is a chemically inert gas that serves to carry the molecules of the analyte through the heated column. The stationary phase is either a solid absorbent, termed gas-solid chromatography (GSC), or a liquid on an inert support, termed gas-liquid chromatography (GLC).</p>  <p style="text-align: center;">Sample injected</p> <p style="text-align: center;">Recorder</p> <p style="text-align: center;">Detector</p> <p style="text-align: center;">Column</p> <p style="text-align: center;">Oven</p> <p style="text-align: center;">Carrier Gas</p>	<p>3 marks for brief explanation of any technique with diagram</p>	
<p>b.)</p>	<p>State the representation of volumetric and gravimetric concentration of gases.</p>		<p>4</p>
<p>Ans:</p>	<p><u>Volumetric concentration:</u> Gas concentration in the atmosphere are represented as parts per million by volume or ppm/v. <u>Gravimetric concentration:</u></p>	<p>2 mark for each</p>	

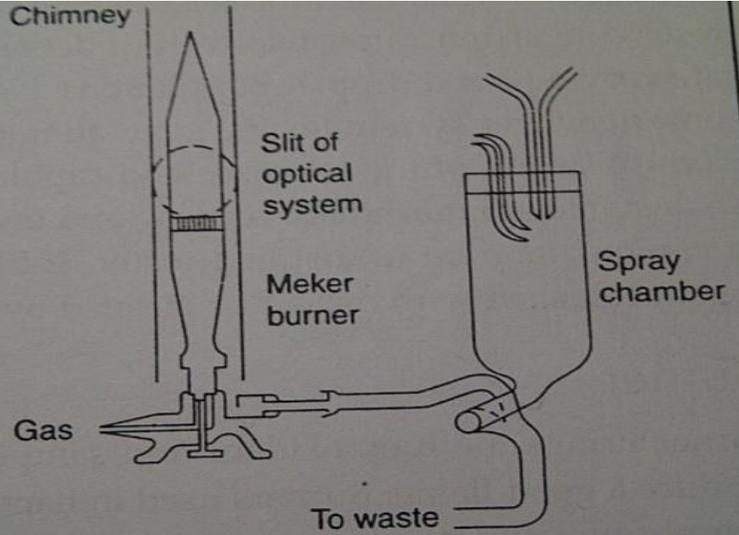


	<p>Toxicological data is represented on Gravimetric basis as $\mu\text{g}/\text{m}^3$ where</p> $\mu\text{g}/\text{m}^3 = \text{ppm} \times \text{PM} / \text{RT} \times 10^3$ <p>P= total atm pressure M= molecular weight of gas R= gas constant T= absolute temperature, K</p>		
<p>c.)</p>	<p>Draw a labeled diagram of Catheter tip electrode for measurement of PO_2 and PCO_2 in blood.</p>		<p>4</p>
<p>Ans:</p>		<p>4 marks for neat labeled digram</p>	
<p>d.)</p>	<p>What is pH? Explain the principle of pH measurement</p>		
<p>Ans:</p>	<p>pH: The pH of a solution is a measure of the molar concentration of hydrogen ions in the solution and as such is a measure of the acidity or basicity of the solution. The letters pH stand for "power of hydrogen" and the numerical value is defined as the negative base 10 logarithm of the molar concentration of hydrogen ions</p>	<p>2 marks for pH</p>	



	<p>Thus pH is defined as the negative logarithm of hydrogen ion concentration in a solution.</p> <p>Or, pH is a logarithmic measurement of the number of moles of hydrogen ions (H^+) per liter of solution.</p> $pH = -\log_{10}[H^+]$ <p>A solution with a low pH value is called an "acid," while one with a high pH is called "alkaline." The common pH scale is a logarithmic scale which extends from 0 (strong acid) to 14 (strong alkaline), with 7 in the middle representing pure water (neutral).</p> <p>Principle of pH measurement</p> <p>The measurement of the pH of a sample can be done by measuring the cell potential of that sample by <i>measurement</i> electrode in reference to a standard <i>reference</i> electrode.</p> <p>The <i>measurement</i> electrode is designed to allow hydrogen ions in the solution to migrate through a selective barrier, producing a measurable potential (voltage) difference proportional to the solution's pH. The circuit will be completed by another electrode called <i>reference</i> electrode. These two electrodes generate a voltage directly proportional to the pH of the solution. At a pH of 7 (neutral), the electrodes will produce 0 volts between them. At a low pH (acid) a voltage will be developed of one polarity, and at a high pH (alkaline) a voltage will be developed of the opposite polarity.</p>	2 marks for principle of pH measurement	
e)	Describe the discharge type atomizer with neat diagram		4
Ans:	Atomizer is the System which is used to form aerosol(fine spray)of the sample by breaking the mass of liquid into small drops in the flame photometer. It introduces liquid sample into the flame at a stable and reproducible rate. It consists of atomization chamber and sprayer. Discharge type atomizer introduces the spray into a condensing chamber and into the flame by the air of the combustible gas air	Explanation 2 marks	



	<p>mixture. It consists of 2 capillary tubes sealed into the walls of a gas chamber, bores are perpendicular to each other, to introduce the spray .The sample solution is atomized by the air from the tip of other capillary. The large droplets of atomized sample condensates and flows down to the waste drain.The smaller droplets are carried by the air stream into the burner, mixed with the burner gases, carried into the region of active combination.</p> 	<p>diagram 2 marks</p>	
<p>f</p>	<p>Explain the term chemical shift with its mathematical expression.</p>		<p>04</p>
<p>Ans:</p>	<p>The difference between the field necessary for resonance in the sample and in some arbitrarily chosen reference compound is called chemical shift.</p> <p>It is the phenomena in which a specification i.e. any atom, in a given molecule resonates at a slightly different frequency based on its local chemical environment. It is the resonant frequency of a nucleus relative to a standard.</p> <p>The variations of nuclear magnetic resonance frequencies of the same kind of nucleus are called the chemical shift. It is the frequency of the resonance expressed with reference to a standard compound.</p> <p style="text-align: center;">OR</p> <p>An NMR spectrum is a plot of the radio frequency applied against absorption or a plot of the intensity of a peak against its chemical shift,</p>	<p>3 marks for chemical shift</p>	



measured in parts per million (ppm). NMR absorptions appear as sharp peaks. A signal in the NMR spectrum is referred to as a resonance and the frequency of the signal is known as its chemical shift.

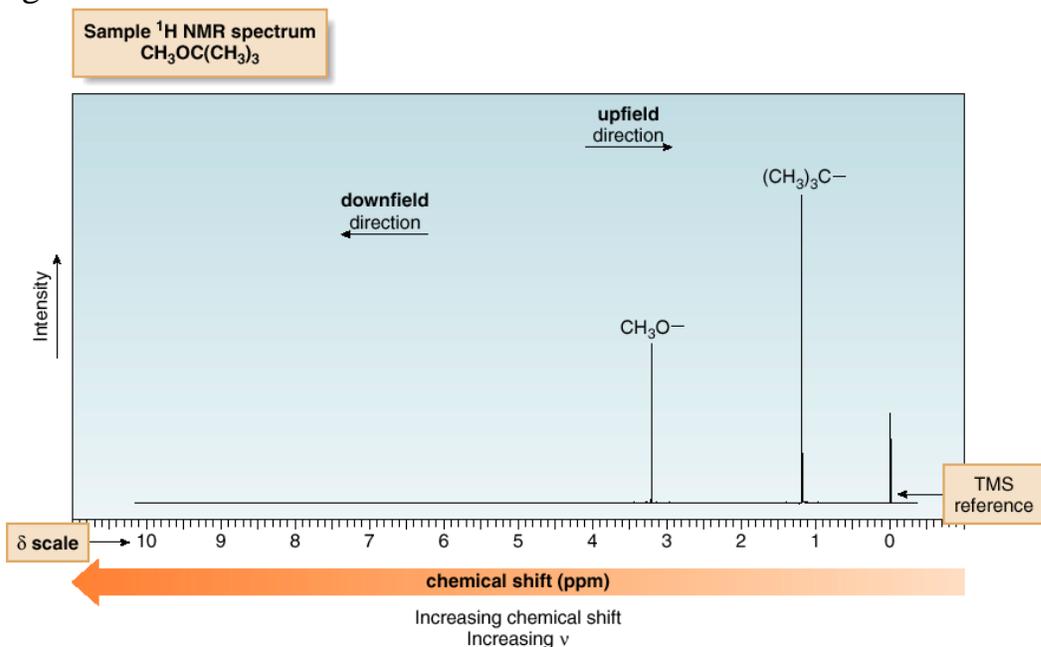
Equation for Chemical Shift δ

$$= \frac{\text{frequency of signal} - \text{frequency of reference}}{\text{Spectrometer frequency}} \times 10^6$$

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

Spectrometer frequency is the radiofrequency.

Chemical shift gives the number of atoms and how they are joined together in molecular



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3.) Attempt any four of the following

16

a.) Draw a neat labeled diagram of double beam densitometer and explain its working.

4

Answer

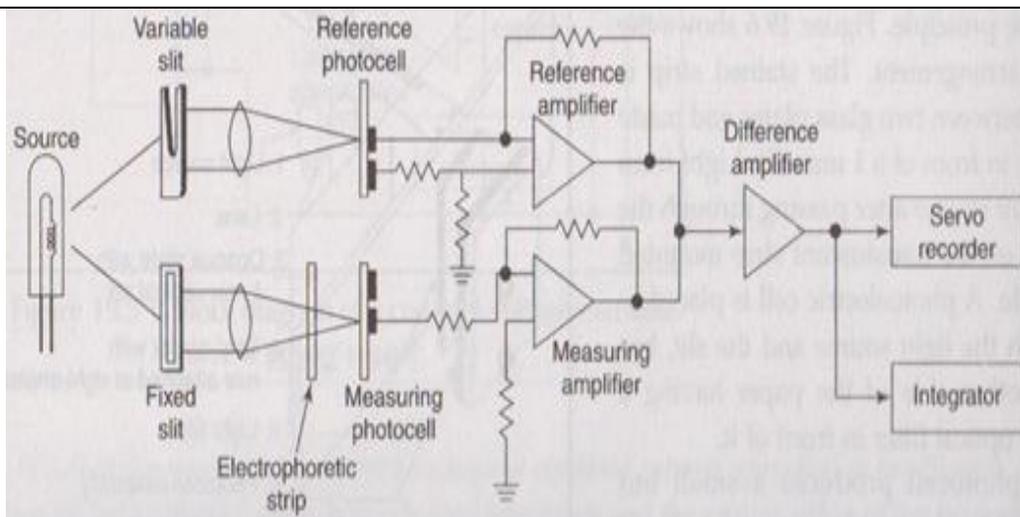


Diagram of Double beam densitometer

Working:

In Double beam densitometer, One photocell act as reference photocell, which receives light directly from the source lamp through a variable slit.

The other photocell called as measuring photocell, receives light through a fixed slit after passing through a stained electrophoretic paper strip.

The photocells are so connected electrically such that net output from them is the difference between the photovoltages of two cells. A DC amplifier is used to amplify this difference to a level sufficient to drive a servo recorder.

Initially, a zero optical density is set on the recorder or meter by allowing the light to pass through the translucent portion of the stained paper placed in front of measuring photocell.

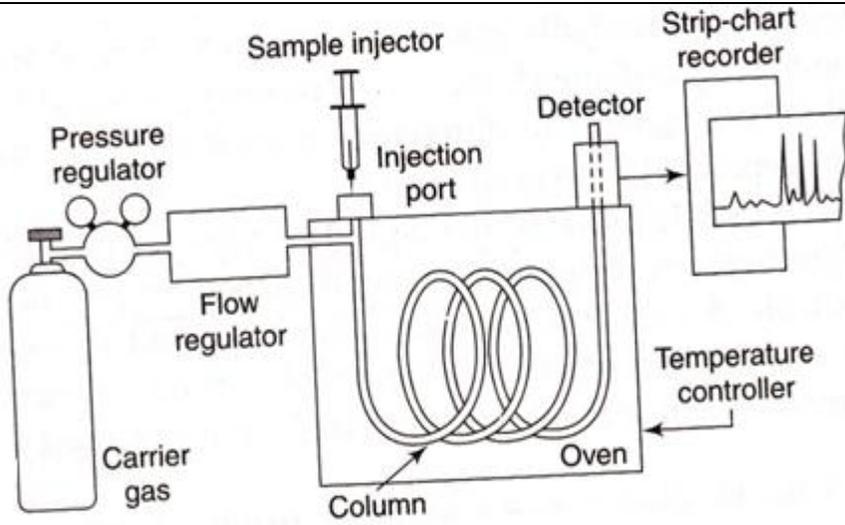
As the stained paper is made to move across the light source, the recorder will trace curves on the graph paper according to the concentrations on the protein samples.

Any fluctuations in the lamp intensity are received simultaneously by both the photocells and effect is automatically nullified.

Some instruments include attachments like electronic integrators for measuring peak height in which results are available directly in concentration.

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for
Diagram**

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for
suitable
explanation**

<p>b.)</p>	<p>Draw a labelled block diagram of gas chromatography and give its working principle</p>		
<p>Ans:</p>	<div data-bbox="316 514 1161 1039" data-label="Diagram">  </div> <p style="text-align: center;"><u>Diagram of gas chromatography</u></p> <p><u>Working principle</u></p> <p>Gas Chromatography is an analytical technique used for compound separation based primarily on their volatilities. It provides qualitative and quantitative information for individually present compounds. Compounds moves through column in gaseous phase and they are portioned between a stationary phases.</p> <p>The career gas (N₂, Ar or He) is available in compressed form in a cylinder fitted with pressure regulator. The gas is conducted through flow regulator to sample injection port maintained at temperature T₁. Gases and liquids samples are injected by syringe. The solute vapour mixes instantaneously with the flowing carrier gas and swept through chromatographic column. It is there the actual separation takes place. Column is maintained at temperature T₂. At the end of the column, solutes emerging individually enter the detectors which produce an electrical signal corresponding to the quantity of solute leaving the column. The detector signal is supplied to recorder and plot of time – signal amplitude called chromatograph is obtained.</p>	<p>2 Marks for Diagra m</p> <p style="text-align: center;">And</p> <p>2 marks for suitable explana tion</p>	



c.)	Give any four applications of NMR	04	
Ans:	<ol style="list-style-type: none">1) NMR is used in magnetic resonance imaging in medical diagnosis2) By studying peak of nuclear magnetic spectra, chemist can determine structure of chemical compound.3) NMR is extremely used for analysis of sample nondestructively.4) NMR is used for data acquisition in petroleum industry and natural gas exploration and recovery.5) NMR is used in process control and process optimization in oil refineries and petrochemical plant.6) It is a complex system integrating several technologies. <p>(Note: marks can be given for any other relevant applications.)</p>	01 mark for each application (any four)	
d.)	Describe the Chemiluminescence technique used for the measurement of nitrogen oxide.	04	
Ans:	<p>The phenomenon of emission of radiation from chemi- excited species is known as Chemiluminescence. It results due to the formation of new chemical bonds. The species in the excited state possess higher energy levels than the ground state and usually have a very short life.</p> <p>Chemiluminescence phenomenon is very useful for measurement of air pollutants, particularly NO and NO₂. Instruments based on the measurement of chemiluminescent emission, based on the following reaction have been developed:</p> $\text{NO} + \text{O}_3 \rightarrow \text{NO}_2 + \text{O}_2$ $\text{NO}_2 \rightarrow \text{NO}_2 + \text{H}\nu \quad (\lambda_{\text{max}} = 6300 \text{ \AA})$ <p>Since NO₂ reacts only slowly with ozone and the reaction which produces NO₃ is not accompanied by Chemiluminescence, it is necessary to reduce NO₂ to NO before admission into the reactor</p> $\text{NO}_2 \rightarrow \text{NO} + \frac{1}{2} \text{O}_2$ <p>Nitric oxide and ozone containing gas steam are mixed in a vessel at a sub atmospheric pressure of about 2 mm of Hg. Light emission is measured with a photomultiplier. With the use of high gain, low dark</p>	04 Marks for relevant explanation	

	<p>current photomultiplier tubes, extremely low levels of radiation can be measured. The response of the instruments based on Chemiluminescence is linear from 1 ppb to 1000ppm of NO. This technique is extremely useful for measurement of NO in automotive exhaust gases</p>		
<p>e.)</p>	<p>Explain the working principle of IR gas analyzer.</p>	<p>04</p>	
<p>Ans:</p>	<div data-bbox="219 724 1242 1459" data-label="Diagram"> <p style="text-align: center;"><u>Diagram for infrared Gas analyzer:</u></p> </div> <p><u>Working :</u> Infrared gas analyzers based on principle that some gases and vapors absorb specific wavelengths of infrared radiation. One of the most commonly measured gases using infrared radiation absorption method is the carbon dioxide. This technique used for this purpose is the conventional doublebeam. One cell is filled with a reference gas, which is a non absorbing gas like nitrogen, whereas the measuring cell contains the sample. The difference is optical absorption detected between the two cells is a measure of absorption of the sample at the</p>	<p>2 Mark for Diagram</p> <p>2 marks for suitable explanation</p>	



	<p>particular wavelength. Since the vibration excitation occurs only if we have hetero-atomic molecules.</p> <p>Infrared analyzers are used for the determination of a large nos of components like CO,CO₂,SO₂,NH₃,H₂O , Nitric oxide as well as most gaseous hydro carbons .</p> <p>One beam passes through the sample cell, and the other beam through a reference cell, and the both beam through a reference cell, and both beams enter opposite ends of the detection chamber. The detection chamber is permanently sealed unit divided into two compartments by a thin, metal diaphragm. Both compartments are charged to the same pressure, with the gas being measured.</p> <p>When the gas being measured enters the sample cell, it absorbs infrared radiation at the same wavelength as gas in the detection chamber. This reduces the amount of radiation reaching the gas in the sample side of the detection chamber and produces a lower pressure in that side .the diaphragm bends toward the sides of lower , and this movement is converted into electrical impulses.</p>		
4 a .)	Attempt any three of the following :		12
i.)	Draw the schematic diagram of time of flight mass spectrometer and explain its working		04

Ans:

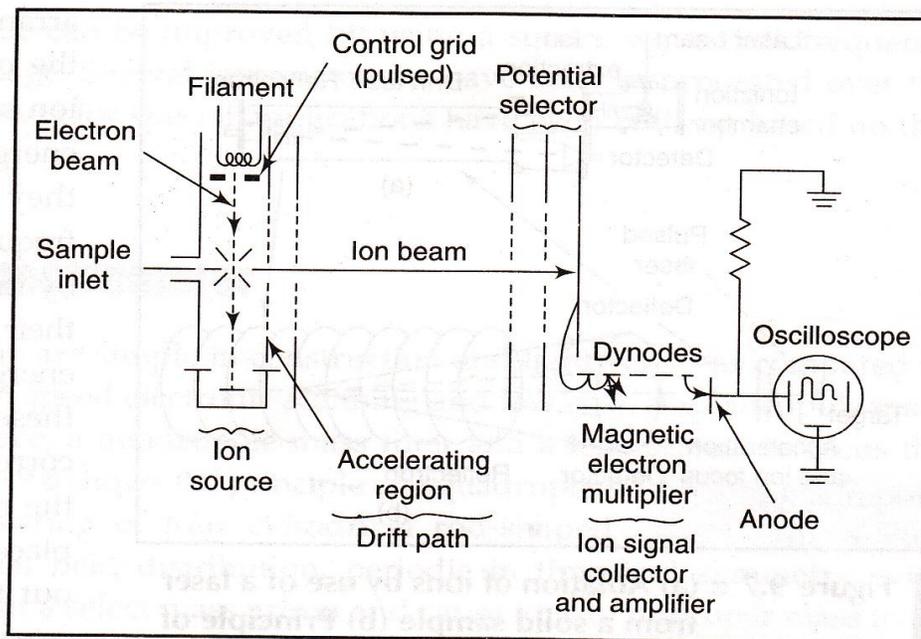


Diagram of time of flight mass spectrometer

Working:

In the time-of flight spectrometer, ions of different mass/ charge ratio are separated by the difference in time they take time to travel over an identical path from the ion source to the collector. This requires the starting time to be well defined. Therefore, ions are either formed by pulsed ionization method or various kinds of rapid electric field switching are used as gates to release the ions from the ion source in a very short time.

In pulse mass spectrometer, ion packets of a few microseconds duration are emitted at intervals of a few milliseconds from a voltage source. The ions transverse an evacuated tube called the drift tube to reach the detector. The detector is sensitized for a brief instant to register their arrival. Since ions of different masses arrive at the detector at different times, the accurate measurement of the time between activating the source and sensitising the detector gives information concerning the mass of the ions. The signal from the ions reaching the detector is amplified and applied to the vertical deflection plates of an oscilloscope. The horizontal axis deflection of the

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Marks
for
Diagram**

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marks
for
suitable
explanation**



	oscilloscope commences as the ion packet start out.This produces a mass spectrum on the screen of the oscilloscope.		
ii.)	How to convert volumetric concentration of gas to gravimetric concentration of gas.		04
Answer	<p>Gas concentrations in atmosphere are generally represented as parts per million by volume, i.e ppm / v or simply ppm per hundred million (pphm), i.e parts per billion (ppb). On the other hand, toxicological data is generally represented on a gravimetric basis , e.g micrograms per cubic meter or milligrams per liter .</p> <p>Conversion from volumetric to gravimetric concentration can be obtained by applying gas laws , the general equation from this being:</p> $\mu\text{g}/\text{m}^3 = \text{ppm} \times \text{PM} / \text{RT} \times 10^3,$ <p>Where ; P= Total pressure (atm) M= Molecular weight of gas of interest R= Gas constant = 0.0821 1-atm /(mole) ($^{\circ}\text{K}$) T=absolute temperature , $^{\circ}\text{K}$</p>	04 marks for relevant explanation	
iii.)	State and explain Beer Lambert’s law.	04	
Answer	<p>Statement: Beer Lambert Law, states the relationship between absorbance (A) and transmittance (T). It states that the concentration of a substance in solution is directly proportional to the ‘absorbance’, A, of the solution.</p> <div style="border: 1px solid black; width: fit-content; margin: 10px auto; padding: 5px;"> $\text{Absorbance } A = \epsilon cb$ </div> <p>Where,</p> <p>A = absorbance (no unit of measurement) E = molar absorptivity ($\text{dm}^3 \text{mol}^{-1} \text{cm}^{-1}$) C = molar concentration (mol dm^{-3}) B = path length (cm).</p> <p>It may be noted that ϵ is a function of wavelength. So, the Beer Lambert Law is true only for light of a single wavelength or monochromatic light. Absorptivity is a constant, depending upon the radiation and nature of the absorbing material. Absorptivity is also sometimes referred to as specific extinction and absorbance as ‘Optical</p>	2 Marks for statement And 2 marks	

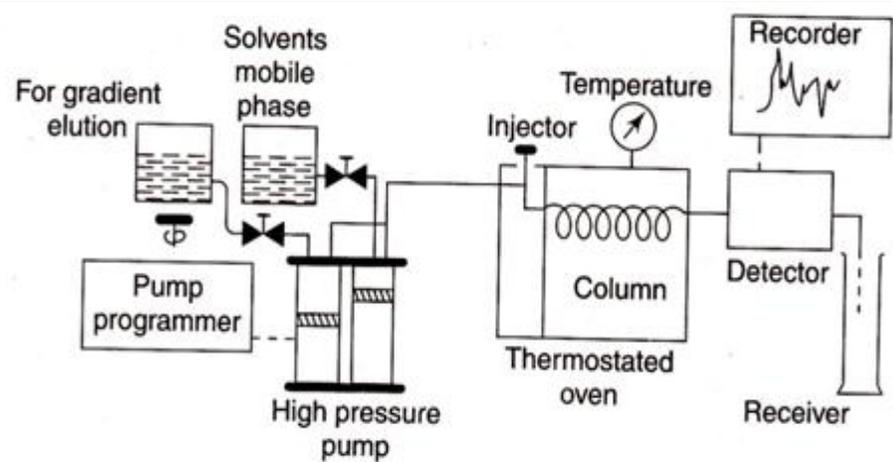


	<p>Density'. Absorbance is the property of a sample, whereas absorptivity is the property of substance and is a constant. Mathematically, absorbance is related to percentage transmittance T by the expression:</p> $A = \log_{10}\left(\frac{I_0}{I}\right) = \log_{10}\left(\frac{100}{T}\right) = \epsilon bc$ <p>The relationship between energy absorption and concentration is of great importance for the purpose of analysis.</p>	<p>for relevant explanation</p>	
<p>iv.)</p>	<p>Explain the constructional details of glass electrode used for pH measurement with the help of a neat schematic diagram</p>		<p>04</p>
<p>Answer</p>	<div data-bbox="430 919 1057 1419" data-label="Diagram"> </div> <p style="text-align: center;"><u>Diagram of Glass electrode</u></p> <p><u>Constructional features:</u></p> <p>Glass electrode consists of thin walled bulb of PH sensitive glass sealed to a stem of non ph sensitive high resistance glass. The PH response is limited to the area of special glass membrane, thus making response independent of depth of immersion. The membrane has thickness of order of 0.05-0.15mm and bulb is of order of 10mm in diameter.</p> <p>The glass pH electrodes are constructed of special glass to create the</p>	<p>2 Marks for Diagram</p> <p>2 marks for relevant explanation</p>	

	<p>ion selective barrier needed to screen out hydrogen ions from all the other ions floating around in the solution.</p> <p>This glass is chemically doped with lithium ions, which is what makes it react electrochemically to hydrogen ions.</p> <p>Since glass is an insulator, this presents a major problem if it is intended to measure voltage between two electrodes. The circuit path from one electrode contact, through the glass barrier, through the solution, to another electrode and back through the other electrode's contact is therefore one of the extremely high resistance.</p> <p>On the inside of membrane is a system of effectively constant pH .it is composed of a silver silver chloride or calomel electrode dipped In hydrochloric acid.Changes in electrical potential on the outer membrane surface are measured by means of an external reference electrode such as calomel and its associated salt bridge.</p>		
<p>4.b</p>	<p>Attempt any one</p>		<p>06</p>
<p>i.)</p>	<p>Describe the working of thermal conductivity gas analyzer with a neat schematic diagram.</p>		<p>06</p>
<p>Ans:</p>		<p>3 Marks for Diagram</p>	



	<p style="text-align: center;"><u>Diagram of thermal conductivity gas analyzer</u></p> <p><u>Working:</u></p> <p>In a typical hot-wire cell thermal conductivity analyzer; four platinum filaments are employed as heat sensing elements. They are arranged in a constant current bridge circuit and each of them is placed in a separate cavity in a brass or stainless steel block. The block acts as a heat sink. The material used for construction of filaments must have a high temperature-coefficient of resistance. The materials generally used for the purpose include tungsten, Kovar (alloy of co,Ni and Fe) or platinum. Two filaments connected in opposite arms of the Wheatstone bridge act as reference arms, whereas the other two filaments are connected in the gas stream, which act as measuring arms. The use of a four-cell arrangement serves to compensate for temperature and power supply variations. Initially, reference gas is made to flow through all the cells and the bridge is balanced precisely with the help of potentiometer D. When the gas stream passes through the measuring pair of filaments, the wires are cooled and there is a corresponding change in the resistance of the filaments. The higher the thermal conductivity of the gas, the lower would be the resistance of the wire and vice versa. Consequently, the greater the difference in thermal conductivities of the reference and sample gas, the greater would be the unbalance of the Wheatstone bridge.</p> <p>(Note: thermal conductivity analyzer using thermistor can be considered)</p>		
ii.)	With a neat diagram explain liquid chromatography .state its application.		06

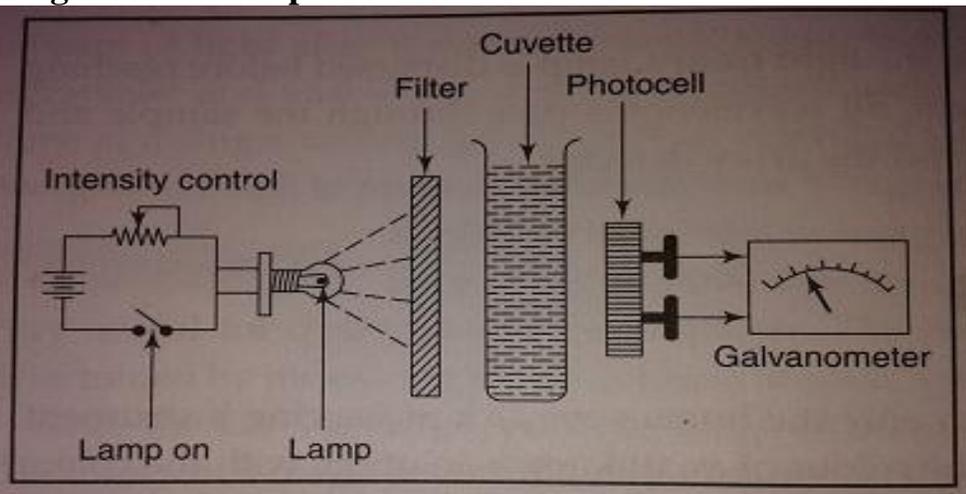
<p>Ans:</p>	 <p style="text-align: center;"><u>Block diagram of Liquid Chromatograph</u></p> <p>It basically consist of :</p> <ul style="list-style-type: none"> (a) A high pressure pump system to force the liquid mobile phase through the column (b) Gradient elution or solvent programmer (c)The sample injection system (d)The column (e)The detection system including display or recording devices (f) Computer for data processing and storage. <p>As in other chromatographic techniques, the sample is introduced into the column with the help of a sample injection system. Various components of the sample are fractionated during their passage through the column. The detection system senses these components as they elute from the column and produces a signal proportional to the amount of solutes passing through the detection system. The detector determines what separation has taken place and provides data permitting qualitative and quantitative evaluation of results.</p> <p>(Note: any other valid liquid chromatography technique can be considered)</p> <p>Applications of liquid chromatography:</p> <p>(1)It is used to separate wide variety of compounds which ranges from</p>	<p style="text-align: center;">2 Marks for Diagram</p> <p style="text-align: center;">And</p> <p style="text-align: center;">3 marks for suitable explain ation</p> <p style="text-align: center;">And</p> <p style="text-align: center;">1 mark for Applica tion (any one)</p>	
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	small organic to large polymer. (2)It uses liquid mobile stage to achieve separation.		
5.	Attempt any four of the following:		16
a)	List the basic component of mass spectrometer and explain any one of them in brief.	04	
Ans:	<p>The following five units are common to most mass spectrometer instruments:</p> <ol style="list-style-type: none">1. The inlet sample system.2. The ion source.3. The electrostatic accelerating system4. The detector, amplifier and display system and5. Auxiliary equipment (pumping system). <p>1.The inlet sample system :</p> <p>a) Gaseous Sample: The introduction of gases merely involves the transfer of sample from gas bulb into metering volumes. The arrangement is small glass manifold of known volume attached to a mercury manometer. The gas sample is introduced into mass spectrometer ion source through a leak of some kind.</p> <p>b) Liquid samples: Liquid samples may be introduced by hypodermic needle and injected through silicon rubber dam or by a break off device which consist in touching a micropipette to sintered glass disc under mercury. The low pressure in the reservoir draws in liquid and vaporizes it instantly.</p> <p>C) Solid Samples: Solid samples can be vaporized to gaseous ion by instantaneous discharge with power up to 100kw by using radiofrequency (1 MHz) spark.</p> <p>2. Ion Source: Following the leak is the ionization chamber which is maintain at low pressure (10^{-4} to 10^{-7} mmHg) and at a temperature of 200 deg Celsius. The electron gun is located perpendicular to the incoming gas stream. Electrons are emitted from filament normally of carbonized tungsten, but for special purposes, tantalum or oxide coated filaments may be used. They are drawn off by a pair of positively</p>	01 Mark for List	
		03 mark for explana tion of any compon ent in brief	



	<p>charged slits, through which they pass into the body of the chamber. The potential presents in the slits controls the electron emission and the energy of the electrons. An electric field applied between these slits accelerates the electrons, which on sub sequent collision with the molecules of the passing gas stream, produced ionization and fragmentation.in order to obtain a mass spectrum, the electric field is kept between 50 and 70V.the electron beam is usually collimated by a magnetic field which is confined to the ionization region.</p> <p>3.Electrostatic accelerating system: positive ions which are separated from electrons by a weak electric field are accelerated into a strong electrostatic field between first and second accelerating slits, voltages of the order of 400-4000V accelerate the ions to their final velocities of up to 150000 miles/s and they acquire a kinetic energy of few thousand electron volts. Such relative high kinetic energy is imparted to the ions to produce an almost monoenergetic beam when it finally emerged out of the final accelerating slit which is approximately 0.076mm in width.</p> <p>4.Ion detectors and recording of mass spectrograph:</p> <p>a) Faraday cup: the ion beam passing through the exit slit of the analyzer tube is normally connected in a cylinder (faraday cage) which is connected to the grid of electrometer tube whose output is in turn amplified. The amplified ion current is recorded as a function of ratio m/e on an oscillograph or pen and ink strip chart recorder. Also following detector and recorders are used</p> <p>b)Channeltron: c)Electron multiplier tube: d)Microchannel plate:</p> <p>5.Auxillary equipment system: In order to prevent undue scattering by collision of ions with residual gas molecules the mass spectrometer requires good vacuum system generally separate mercury or oil diffusion pumps are employed in the source and analyzing regions of spectrometer. Generally mechanical pump, diffusion pump, turbomolecular pumps are used.</p>		
b)	State the principle of colorimeter. Explain the working of single beam filter photometer with neat labeled diagram.	04	

<p>Ans:</p>	<p><u>Working Principle:</u></p> <p>Colorimetric methods are more precise and eliminate the necessity of preparing a series of standards every time a series of unknowns is run. Colorimetric determination involves visual measurement of colour and this method employ photoelectric measurement hence called as photometric or spectrophotometric method. As this method involves measurement of color in electromagnetic spectrum (400-700mμ) thus is referred as colorimetric method.</p> <p>In a colorimeter sample is normally a liquid. The sample compartment of colorimeter is provided with a holder to contain the cuvette in which liquid is examined. The holder is mounted on the slide with positions for at least two cuvettes so that sample and reference cuvettes are measured first and a shutter is moved in to or out of light beam until the micro ammeter gives full scale deflection. The sample is then moved into the beam and the light passing through it is measured as a percentage of reference value.</p> <p>Sample concentration = Standard concentration * (Sample reading /reference reading)</p> <p>Single beam filter photometer:</p>  <p>The figure shows basic component of filter photometer.</p>	<p>01 mark for principle</p> <p>02 Marks for diagram</p> <p>& 01 for explanation</p>	
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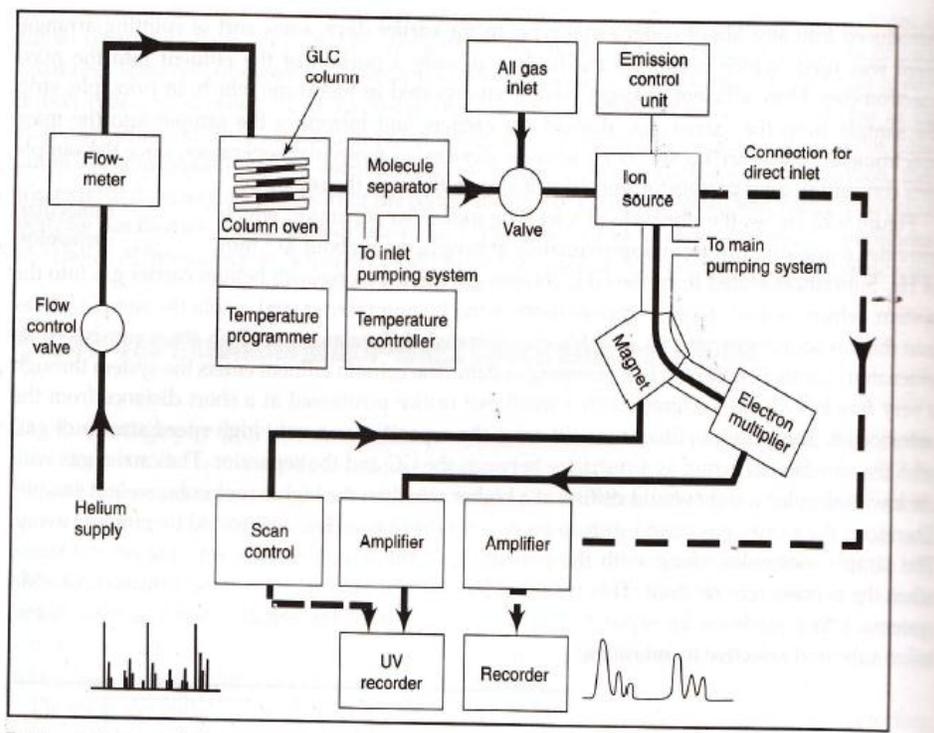
	<ol style="list-style-type: none">1. The source of light is tungsten filament lamp which is held in a reflector and which through light on the sample holder through a filter.2. The filter may be either of absorption or interference type.3. The sample holder is a cuvette with parallel walls or may be a test tube.4. The light after passing through the sample holder falls on the surface of photocell. The output of cell is measured on micro ammeter. In order to operate instrument following steps are taken<ol style="list-style-type: none">1. With photocell darkened the meter is adjusted mechanically to read zero.2. The blank or pure solvent or reference solution is inserted in the path of light beam and incident light intensity is regulated.3. Solution of both standard and unknown are inserted in the place of blank and the reading of specimen relative to blank is recorded. The meter scale is calibrated in linear transmittance unit.		
c)	Why the column temperature is so critical in gas chromatograph?	04	
Ans:	<p>The columns of gas chromatography is not normally operated at room temperature because it would then be suitable only for the analysis of gases and extremely volatile liquid therefore it must be heated in some form of thermostat. moreover it is desirable to keep column at precisely constant temperature. This is essential because the quantitative response of the detector is often affected by the column temperature for this purpose the column is housed in an oven. The oven is thermally insulated so that heat losses to the atmosphere is minimized. Accurate temperature is chosen that gives satisfactory time for analysis Now when the column temperature is kept constant it is difficult to analyze samples having components of a wide boiling range this difficulty can be overcome by using program heating of the column so that its temperature is not kept constant but is subjected to an exactly control temperature rise while separation is in progress Low temperature is beneficially for better separation of low boiling point components and high temperature more rapid elution of high boiling points components thereby shortening the time of analysis and sharpening the resultant chromatographic peaks.</p>	04 Marks for suitable explanation	



	Thus the column temperature is a critical factor in gas chromatography.		
d)	Explain the gas chromatography technique for the measurement of Carbon monoxide in air.	04	
Answer	<p>1. When an air sample containing CO is passed through a stripper column, the heavy hydrocarbons are retained and CO and methane are passed into chromatographic column and then into a catalytic reducing chamber.</p> <p>2. The methane would pass through the reducing chamber unaffected while CO is reduced to methane.</p> <p>3. By using hydrogen flame ionization detector both methane peaks can be detected.</p> <p>4. The first peak is due to methane while the second peak would correspond to CO.</p> <p>5. The accuracy is about +-2%. peak heights of CO and CH₄ would give sensitivity of 50 ppb.</p>	04 Mark for relevan t explana tion	
e)	Explain the principle of electrophoresis. State its application(Any two)	04	
Answer	<p>Electrophoresis:</p> <p>Electrophoresis is a separation technique that is based on the mobility of ions in an electric field. Positively charged ions migrate towards a negative electrode and negatively charged ions migrate toward a positive electrode. For safety reasons, one electrode is usually at ground and the other is biased positively or negatively. Ions have different migration rates depending upon their total charge, size and shape, and can therefore be separated.</p> <p>The moving boundary method of electrophoresis utilizes the migration of the particles in free solution and observation of various molecular boundaries through sensitive refractometric techniques. With this the value of electrophoresis in obtaining distinct and measurable fractions of variety of substances got well established particular in clinical laboratories.</p> <p>Basically the electrophoresis technique separates the molecules based on size and charge under the influence of electric field. If E is a strength of electric field is a charge on molecule and F is</p>	02 Marks principl e	



	<p>the frictional force on the molecules then V the velocity of migration is given by $V = EZ/F$ The frictional force can be defined as $F = 6\pi\eta r$ Where η is viscosity of medium and r is stoke radius of molecules therefore $V = EZ/6\pi\eta r$ This implies that the electrophoretic mobility is proportional to the charge on molecules and inversely proportional to the radius of molecules.</p> <p>Applications of Electrophoresis:</p> <ol style="list-style-type: none">1. It is used in the separation of amines, amino acids, phenols etc.2. It is used in the separation and identification of highly complex protein mixture.3. It is used in the separation of enzymes.		
f)	Draw a labeled block diagram of GCMS	04	
Ans:	Diagram of GCMS:	04 marks for neat labeled diagram	



6. Attempt any Four of the following :

16

a) State the applications of flame photometry.(any four)

04

Answer Applications of flame photometry:

1. It is used in determination of potassium, sodium, magnesium and calcium in biological fluids like serum, plasma, urine etc, is routinely carried out by flame photometer.
2. Analysis of industrial water, natural water for determining elements responsible for hard water (magnesium, barium, calcium etc.) is standard procedure in many laboratories.
3. Soil samples are routinely analyzed mainly for sodium and potassium and also for calcium and magnesium (after removing other interfering elements) by flame photometer.
4. Some important elements which are commonly determined by this method are aluminum, barium, calcium, chromium, copper, iron, lead , magnesium, potassium, zinc, manganese, and

01
Marks
for
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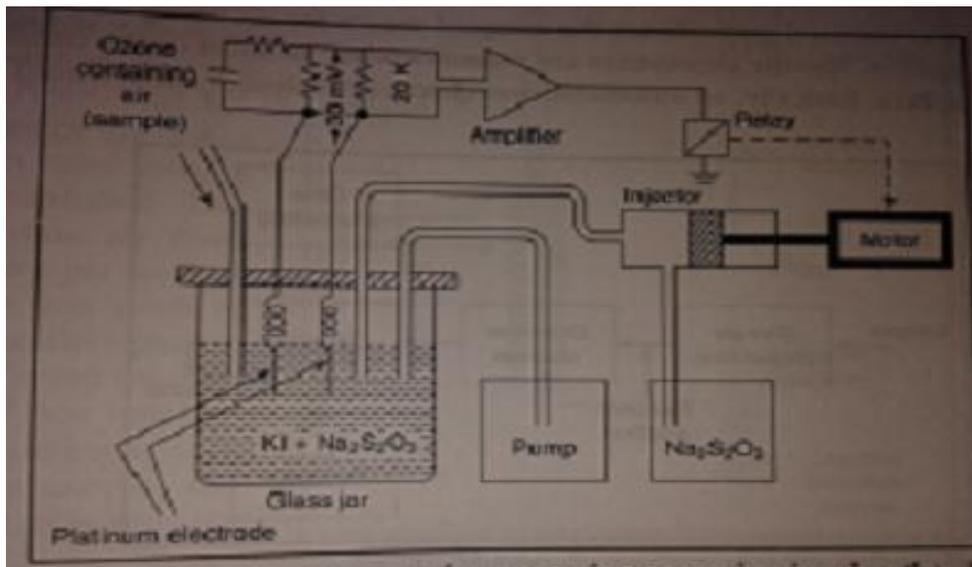
	<p>strontium.</p> <ol style="list-style-type: none">In glass industry, flame photometry is used in determining of sodium, potassium, boron, lithium etc.In cement industry, this method is used in estimation of sodium, potassium, calcium, magnesium, manganese, as well as lithium.Flame photometry is extensively used in estimation of alkali-alkaline earth metals as well as other metals present in metallurgical products, catalysts, alloys etc.Flame photometry has also been used in determination of certain metals like lead, manganese, in petroleum products like gasoline, lubricating oils and organic solvents.Analysis of ash by flame photometer is routinely carried out in various industries for estimating alkali and alkaline earth metals as their oxides.		
b)	State the principle of NMR. Explain the resonant condition of NMR.	04	
Answer	<p>Principle of NMR: Nuclear Spin: Elementary particles such as electrons or a nucleus behaves as if they rotate about an axis possesses the property of spin known as nuclear spin. The angular momentum is associated with the spin of particle would be an integral or half integral multiple of $h/2\pi$ where, h is planck's constant. Nuclear energy level : Since a nucleus possesses a charge, its spin gives rise to a magnetic field that is analogues to the field produced when an electric current is passed through a coil of wire. The resulting magnetic dipole or nuclear magnetic moment μ is oriented along the axis of spin and has a value that is characteristic for each kind of particle. When spinning nucleus is placed in a strong uniform magnetic field (H), the field exerts a torque upon the nuclear magnet. This would make the nucleus to assume a definite orientation with respect to the external field. The torque is a vector with its direction at right angles to the plane of μ and H. This results in a rotation of the nuclear axis around the direction of the external field. This is called precessional</p>	02 mark for principle	



	<p>motion.</p> <p>Resonant Condition:</p> <p>When an alternating RF field, superimposed over the stationary magnetic field, rotates at exactly the frequency of an energy level, the nuclei will be provided enough energy to undergo a transition from lower energy level to a higher energy level. In general Energy difference between states is given by,</p> $\Delta E = \mu\beta.H_o/I$ <p>Where,</p> <p>H_o = strength of external magnetic field in gauges B = constant called the nuclear magneton, 5.049×10^{-24} ergs μ = magnetic moment of the particle expressed in units of nuclear magnetons</p> <p>The frequency, ν of radiation determine from Planck's equations</p> $\Delta E = h\nu = \mu\beta.H_o/I$ <p>The frequency can be varied by applying magnetic field,</p> $\begin{aligned} \nu &= \mu /h.\beta. H_o/I \quad \dots h = 6.626 \times 10^{-27} \text{ ergs} \\ &= \frac{2.797 \times 5.05 \times 10^{-24} \times 23000}{(6.6256 \times 10^{-27}) (1/2)} \\ &= 95 \times 10^6 \text{ Hz} \\ &= 95 \text{ MHz} \end{aligned}$ <p>The proton will process 95 million terms per second in a field of 23000 gauss. The frequency 95 MHz lies in the radio frequency range of the electromagnetic spectrum.</p>	<p>02 mark for Resona nt Condi on in brief</p>	
<p>c)</p>	<p>Draw the neat labeled block diagram of complete blood gas analyzer.</p>	<p>04</p>	

<p>Answer</p>	<p>Diagram of complete blood gas analyzer:</p>	<p>04 mark for Neat labeled Diagram</p>	
<p>d)</p>	<p>Describe the ozone measurement using conductivity metry with the help of neat schematic diagram</p>	<p>04</p>	
<p>Ans:</p>	<p>A wet chemical method which uses the oxidizing properties of O₃ is employed to sensitive meter for continuous sampling of contaminating oxidants in atmosphere. The ozone containing air is bubbled into potassium iodide solution and resulting iodine determined by measuring current through the cell. The current is related to ambient O₃ levels by previous calibration with known ozone source. Thus construct air-ozone meter which measures and records instantaneous</p>	<p>02 Marks for Diagram</p>	

ozone concentrations.

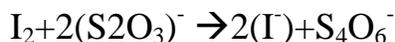


The arrangement is as shown in figure. It consist of an hermetically sealed glass jar containing 150 cm³ of buffered 10 % KI solution and 0.5 cm³ sodium thiosuphate of known concentration. Two spiral platinum electrodes dip into the solution and bias voltage of 30mV is applied across them. The air above the solution is evacuated, whereas the outside is let in through a Tygon tube which is inert to ozone.

When ozone enters the solution the following reaction takes place



The iodine then reacts with thiosuphate

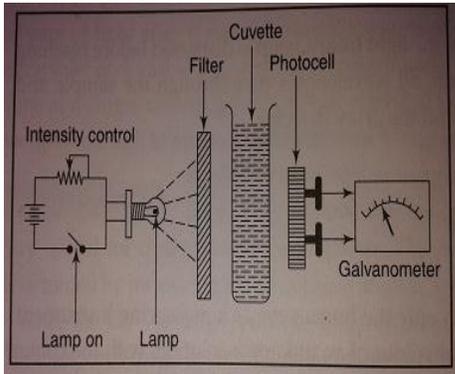
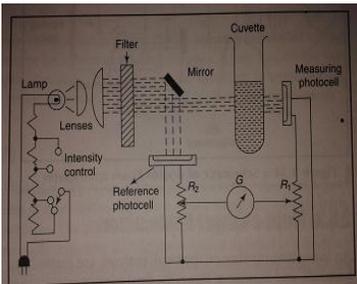
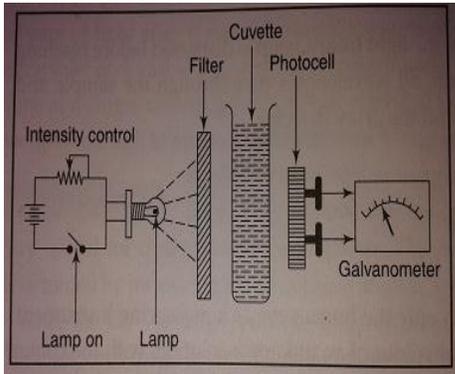
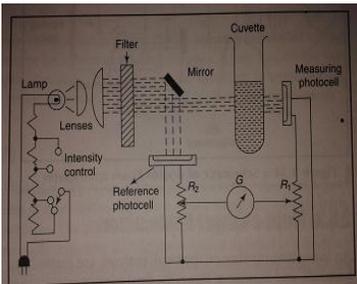
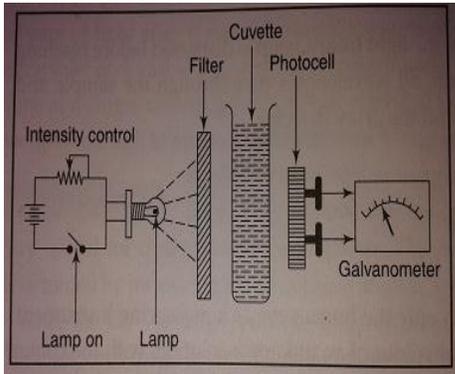


Reaction continues so long as there is thiosuphate in the solution. When all the thiosuphate has been reacted, free iodine appears and reacts at the electrodes.

The electrical resistance is high as long as there is an excess of thiosulphate. The resistance decreases when it is used up. This change is used to control the operation of instrument. The voltage drop across 20kohm resistance which is in series with the electrode is used to operate the recorder as well as relay which controls a motorized injector which injects 0.5cm³ thiosulphate in each operation. The recorder serves mainly to indicate as to when the injection was made

**02 for
Explan
ation**



	and thus the average ozone concentration between any two injections can be calculated. Since the pumping rate is known, knowledge of the time intervals gives the total volumes of air sample.						
e)	Compare single beam and double beam filter photometer	04					
Ans:	<table border="1"> <thead> <tr> <th>Single Beam Filter Photometer</th> <th>Double Beam Filter Photometer</th> </tr> </thead> <tbody> <tr> <td> 1. Reading error due fluctuation in lamp intensity. 2. Single photocell is employed. 3. Light pass through sample & test solution on single photo cell. 4.  </td> <td> 1. Error due to lamp intensity is minimized. 2. Two photocells are normally employed. 3. Light is split through test solution on measuring photocell & other directly on reference photo cell. 4.  </td> </tr> </tbody> </table>	Single Beam Filter Photometer	Double Beam Filter Photometer	1. Reading error due fluctuation in lamp intensity. 2. Single photocell is employed. 3. Light pass through sample & test solution on single photo cell. 4. 	1. Error due to lamp intensity is minimized. 2. Two photocells are normally employed. 3. Light is split through test solution on measuring photocell & other directly on reference photo cell. 4. 	1	Marks for each Points
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