(Autonomous)

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SUMMER - 2017 EXAMINATION

Model Answer

Important Instructions to examiners:

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- 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 judgement 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 & its Answer	Remark	Total Marks
01 A)	Attempt any THREE		12
a)	Describe the working principle of a mass Spectrometer with suitable diagram.	04	
Ans.	Diagram mass Spectrometer: A. Sample B. Ions C. Magnetic field D. Circular path of deflected ions E. Collector electrode F. Slit Recorder Amplifier	02 marks for diagram	
	Principle of operation of a mass spectrometer: The molecules in the gas sample (A) to be analyzed are bombarded with electrons to produce ions (B). These ions are accelerated in a high vacuum into a magnetic field (C), which deflects them into circular paths (D). Since the deflection for light ions is greater than that for heavy ions, the ion stream separates into beams of different molecular weight. A suitably placed slit (F) allows a beam of a selected molecular weight to pass through to a	02 marks for	



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d) Ans.	R= Gas constant = 0.0821 1-atm /(mole) (°K) T=absolute temperature, °K Differentiate between photometer & spectrophotometer(any 4 points).	04 01 mark	
1)	T=absolute temperature, °K	0.4	
	$ID = C_{oc}$ constant $= IIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIII$		
	M= Molecular weight of gas of interest		
	P= Total pressure (atm) M= Molecular weight of gas of interest		
	Where;		
	$\mu g/m^3 = ppm \times PM / RT \times 10^3,$		
	obtained by applying gas laws, the general equation from this being:		
	Conversion from volumetric to gravimetric concentration can be	ion	
	gravimetric basis, e.g. micrograms per cubic meter or milligrams per litre.	explanat	
	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	marks for	
Ans.	Gas concentrations in atmosphere are generally represented as parts per million	04	
	concentration of gas.		
c)	How to convert volumetric concentration of gas to gravimetric	04	
	Any other relevant uses shall be considered		
	OR		
	5) To standardize the electrode chains for pH measurements.		
	4) To create and maintain desired stabilized pH in a solution		
	dihydrogen phosphate with 0.025 molar disodium hydrogen phosphate and 0.01 molar potassium dihydrogen phosphate with 0.04 molar disodium hydrogen phosphate.	four)	
	3) Buffer solutions used in blood pH measurement: 0.025 molar potassium	use(any	
	2) Buffer solutions are characterized by their pH value.	for each	
	hydrogen ions.	mark	
	counteracting pH changes in the solution as caused by the addition or removal of	01	
Ans.	1)Buffer is a substance which by its presence in a solution is capable of	U T	
b)	State the use of buffer solution in blood pH measurement.	04	
	indicate the number of ions in each beam. The proportion of molecules of different masses in the gas sample may thus be found and a complete analysis of the gas sample may be made; provided all the constituent gases have a different molecular weight. This is usually the case in respiratory gas analysis work.		
	collector electrode (E). As the accelerating voltage on the ion source is gradually reduced, ion beams of successively greater mass pass through the slit. When these ions fall on the collector electrodes, they produce minute electric currents, which may be measured after suitable amplification. Their amplitude will	relevant explanat ion	



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	Photometer	Spectrophotometer	for each	
	Photometer measures intensity	Spectrophotometer measures	point	
	of light.	intensity of light at different	(any	
		wavelengths.	four	
	Photometer is simpler to design.	Spectrophotometer is complex	points)	
		to design.		
	The amount of light reaching the	The amount of light reaching the		
	detector is more than	detector is much smaller than		
	spectrophotometer Photometer uses colour filter	photometer.		
	Photometer uses colour filter	Spectrophotometer uses monochromater		
	Agourgay is law			
	Accuracy is low	Accuracy is high OR		
	(Any relevant 04 points sh			
	(Any relevant 04 points sn	an be considered.)		
)1	Attempt any ONE			06
3)	Attempt any ONE			00
a)	Explain with neat diagram basic	elements of gas chromatography.	06	
Ans.	Zipium with neut ungrum suste	ordinario or gas em omatography.	03	
	250	Strip-chart	marks	
	Sample in	njector recorder	for	
	#	Detector	diagram	
	Pressure	Injection		
	regulator	port		
		Part Part Part Part Part Part Part Part		
	Flow			
	regulator	(((())(())) Temperature		
		controller		
	1.1			
	Carrier	Oven		
	gas	olumn		
	Diagram of	gas chromatography		
	Working principle:		03	
		ical technique used for compound separation	marks	
		ies. It provides qualitative and quantitative	for	
		ent compounds. Compounds moves through	explanat	
		are portioned between a stationary phases.	ion	
	The career gas (N2, Ar or He) is	available in compressed form in a cylinder		
		e gas is conducted through flow regulator to		
	_	at temperature T1. Gases and liquids samples		
		ute vapour mixes instantaneously with the		1



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b)	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. Draw block diagram of Analytical instrument & explain function of each block.	06	
Ans.	Elements of an analytical instrument Explanation of Analytical Instrument: Analytical instruments provide information on the composition of a sample of matter. The basic block diagrams are, 1. Chemical information source: Chemical information source which generates a set of signals containing necessary information. The source may be in the sample itself. For example yellow radiation emitted by heated sodium atoms constitutes the source of the signal in a flame photometer. 2. Transducer: Transducer which converts the nature of the signal Because of the familiar advantages of electric and electronic methods of measurement, it is the usual practice to convert all non-electrical quantities. Associated with the analysis of a sample into electrical form. 3. Signal conditioner: Signal conditioner which converts the output of the transducer into electrical quantity suitable for operation of display system. Signal conditioner may be varying in complexity from a simple resistance network or impedance matching device to multi-stage amplifiers and other electronic circuit. 4. Display system: It provides a visible representation of quantity as displacement on a scale or on the chart of a recorder, CRT.	02 marks for block diagra m	
02	Attempt any FOUR		16
a)	State & explain Beer Lambert's law	04	
Ans.	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. Absorbance $A = \varepsilon$ cb Where,	02 Marks for stateme nt	



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	A = absorbance (no unit of measure	ement)			
	$\varepsilon = \text{molar absorptivity (dm3 mol -1)}$,			
	C = molar concentration (mol dm -:				
	B = path length (cm).				
		of wavelength. So, the Beer Lamber	t Law is		
		elength or monochromatic light. Abs			
	is a constant, depending upon the r	adiation and nature of the absorbing i	material.		
	Absorptivity is also sometimes refe	erred to as specific extinction and abs	sorbance	02	
	as 'Optical Density'. Absorbance	e is the property of a sample,	whereas	marks	
	absorptivity is the property of subst	ance and is a constant.		for	
	Mathematically, absorbance is re-	lated to percentage transmittance T	by the	Explana	
	expression:			tion	
	$A =) =) = \varepsilon bc$				
		absorption and concentration is	of great		
	importance for the purpose of analy	vsis.			
b)	Give the comparison between	een gas chromatography &	Liquid	04	
	chromatography. (Any 4 pts.)				
Ans.				01 mark	
	Gas chromatography	Liquid chromatography		for each	
	In this mobile phase gas is used	In this mobile phase liquid is		point	
		used		(any	
	Types are gas/liquid & gas/solid	Types are paper, column and		four	
		thin layer.		points)	
	Detectors used are thermal	Detectors used are UV			
	conductivity detector , flame	spectrophotometric detector ,			
	ionized detector , flame	florescence detector, RI detector			
	photometric detector etc.				
	He, Ar, Ne gases may be. Used	Volatile solvents, Eluent			
	as carrier.	solution are used as carrier.			
	Accuracy is more	Accuracy is less			
	Technique is simple	Technique is complicated			
	Takes less time	Takes more time			
		OR			
	(Any relevant 04	points shall be considered.)			
c)	Describe measurement technique	s for SO ₂ using conductivity meter.		04	



Expansion chamber

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02 Ans. To reagent reservoir marks Filter diagram C

To waste

Conductivity method for measurement of SO₂ in air :

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.

 $H_2O_2+SO_2 \rightarrow H_2SO_4 \rightarrow H^+ + (HSO4)^-$

Capillary

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 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 SO2 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 SO₂ at any instant is proportional to slope of saw-tooth at that instant.

d) Describe principle of operation of Nuclear Magnetic Resonance Spectroscopy

Nuclear Magnetic Resonance spectroscopy is a non-destructive technique for mapping molecular structures and learning how molecules function and relate to each other. It works on the principle of Nuclear Magnetic Resonance, which is study of absorption of radiofrequency radiation by nuclei in a magnetic field.

1. The utility of NMR spectroscopy for structural characterization arises because different atoms in a molecule experience slightly different 02 marks **Explana** tion

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04

04

for

ion

marks

explanat

Ans.



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	,	<u> </u>
	magnetic fields and therefore, transitions at slightly different resonant frequencies in an NMR spectrum. 2. Splitting of the spectra line arises due to interactions between different nuclei, which provide information about the proximity of different atoms in a molecule. Write four types of gas pollutants with their typical concentration valves.	04
e)	Write four types of gas pollutants with their typical concentration values.	-
Ans.	The major gas pollutants are carbon monoxide, sulphur oxides, hydrocarbons, nitrogen oxides and particulates. Carbon monoxide: Its average concentration is below 200 ppm.	01 mark for each type with
	Hydrocarbon: Its average concentration is below 80 ppm	typical concent ration
	Oxidants: Its average concentration is below 500 ppb	values
	Sulphur dioxide: Its concentration in urban areas is 0.024 ppm.	
	Nitrogen oxides: Its level ranges from 0.5 to 0.12 ppm.	
f)	Draw & Explain with neat diagram working of IR gas analyzer.	04
Ans.	Chopper motor Infrared source Calibrated meter Chopper Test gas Integrator Working:	Marks for diagram
	Infrared gas analyzers based on principle that some gases and vapours 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 double-beam. 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 particular wavelength. Since the vibration	02 marks for relevant explanat ion



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_			
	excitation occurs only if we have hetero-atomic molecules.		
	Infrared analyzers are used for the determination of a large no of components like CO, CO ₂ , SO ₂ , NH ₃ , H ₂ O, Nitric oxide as well as most gaseous hydro carbons. 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. 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.		
03	Attempt any FOUR		16
a)	Describe operation of discharge type Atomizer used in flame photometer with neat diagram.	04	
Ans.	Figure shows discharge-type atomizer. It consists of two capillary tubes sealed into the walls of glass chamber in such a manner that their bores are perpendicular to each other. The sample solution is poured into a funnel or drowns up from a container and is atomized by the blast of air from the tip of the other capillary. Chimney Chimney Spray Chamber Chimney Spray Chamber However, the atomized stream is composed of coarse spray with large droplets, which condense on the walls of the chamber and helical tube leading to the burner. The condensate flows down to the waste drain. The smaller droplets, in the form of virtual fog, are carried by the air stream into the burner, where the condensate is mixed with the burner gasses and carried it to the region of active combination. Two renewable hypodermic needles of stainless steel or glass are	02 Marks for diagram 02 marks for relevant explanat ion	



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b)	comparatively high and ranges between 4 and 25 ml of solution per minute. Of this amount, only 5% actually reaches the flame. The sensitivity of this type of atomizer can be markedly increased by using a chamber which is heated by an electric heater placed around its walls. This hastens the process of vaporization of the solvent and produces an aerosol of very fine particles, all of which are swept into the burner. In this case, sample consumption is only 0.2 to 0.6 ml per minute and a substantial portion is carried directly into the burner to yield a much higher sensitivity. What is Resonance Condition? Describe Nuclear energy level in NMR	04	
D)	spectrometer.	04	
Ans.	Resonant Condition: 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, $\Delta E = \mu \beta. Ho/I$ Where, $Ho = \text{strength of external magnetic field in gauges}$ $B = \text{constant called the nuclear magneton, } 5.049 \times 10\text{-}24 \text{ ergs}$ $\mu = \text{magnetic moment of the particle expressed in units of nuclear magnetons}$ 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	02 Marks for conditio n 02 marks for descript ion	
	rotation of the nuclear axis around the direction of the external field. This is called processional motion.		
c)	Describe construction & working of calomel electrode used for pH measurement.	04	
Ans.	Diagram of Calomel Electrode:	02 marks for diagram	



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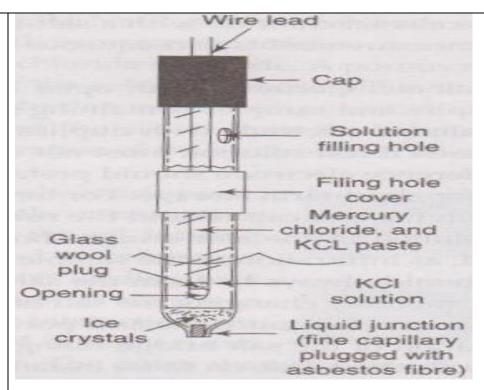
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Model Answer



Calomel Electrode:

The purpose of the calomel or reference electrode is to provide stable, reproducible voltage to which the working (detector) electrode potential may referenced. The most common reference electrodes which meet these criteria are: Mercury/mercurous electrode (calomel), and Silver/silver chloride electrode (Ag/AgCl) electrode.

Therefore, in order to measure the potential changes of the pH – sensitive electrode directly, it is of a stable reference electrode, whose potential remains unaffected by changes in the composition of the cell solution. The reference electrode against which the potential of the glass electrode is measured is the calomel electrode.

As shown in the figure

- 1. Calomel electrode consist of, a metallic internal element, typically of mercury-mercurous chloride (calomel), immersed in an electrolyte, which is usually a saturated solution of potassium chloride.
- 2. The electrolyte solution forms a conductive salt bridge between the metallic element and the sample solution, in which the measuring and reference electrodes are placed.
- 3. For stable electrical conductivity between the internal metallic element

02 for explanat ion



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and sample solution, a small but constant flow of electrolyte solution is maintained through a liquid junction in the tip of outer body of reference electrode. 4. $\frac{1}{2}$ Hg₂Cl₂ + e $\leftarrow \rightarrow$ Hg + Cl⁻ 5. Since at given temperature, the activity of mercurous chloride is constant and that of the mercury is unity by definition, it is chloride ion activity which is potential determining. The most common source of chloride ion is potassium chloride saturated at 3.8, 3.5 or 0.1 M concentrations. d) **Describe** briefly significance of chromatographic column 04 used in chromatography 1. The column is the main part of a gas chromatograph, where the 04 Ans. marks fundamental process of separation takes place. for explanat 2. It operates on the fact that a sample of a gas or vapour is introduced into ion a column, it spreads by a molecular diffusion to get a concentration profile. 3. As the sample moves through the column, additional spread takes place, but the band maintains its general shape, which is detected and recorded as the familiar chromatographic peak. 4. The degree of peak broadening with respect to time and column length is an indication of column efficiency. 5. Column performance is usually measured by the number of theoretical plates, which may be determined from the dimensions of peaks. 04 e) Describe how measurement of Nitrogen oxide is done using CO laser. 02 Ans. marks Sample Infrared detector for diagram Gas transfer amplifier Recorder Nitrogen oxides measurement using CO laser: 1. Figure shows the block diagram of detecting nitric oxide in 0.25 ppm concentration. 02 2. Apparatus consists of CO Laser, which emits radiation that is absorbed marks by the NO in the mixture. for



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	 The amount of absorption being proportional to the concentration of NO present. The CO laser used is a dc excited continuous working laser, which operates on a single wavelength of 5.307 μ and at liquid nitrogen temperature. A diffraction grating is used at one end of the cavity as a selector. The absorption cell is made of pyrene and is of 15 mm diameter and 90cm length. It is evacuated to a press of 10- 6to 10-5. The DC Magnetic field produces a field up to 2.5 KG by a solenoid. The detector is a liquid nitrogen cooled Ge-Au element. The signal is amplified and locks in an amplifier before given to recorder. The signal amplitude varies linearly with the concentration of NO in the sample. 	Explana tion	
04	Attempt any THREE		12
(A) (a)	With a block diagram, explain working of liquid chromatography.	04	
Ans.	Block diagram of liquid chromatography	02	
	Solvents	marks	
	For gradient mobile Temperature	for	
	Pump programmer Thermostated oven Receiver pump	diagram	
	It basically consist of:		
	(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. 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.	02 Marks for Explana tion	



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Draw and labelled diagram of catheter tip electrode for measurement of b) 04 **PO₂ & PCO₂ in blood gas analyzer.** Gelled 02 Ans. electrolyte Silver/silver chloride Diffusion marks internal reference membrane for pH glass diagram Semi-solid electrolyte Silver cathode Silver/silver chloride common reference electrode Silicon rubber Catheter Lead out OR (Any other relevant diagram shall be considered) The partial pressure of oxygen in blood or plasma indicates the extent of oxygen between lungs and blood. Catheter tip electrode is small enough to be mounted on Catheter tip and preferred for measurement of more than one parameter. 02 Catheter tip electrode is used for continuous measurement PO2 and PCo2. **Marks** A180Um diameter silver cathode constitutes a po2 measuring electrode. The for **Explana** common electrode used is silver/silver chloride electrode. A semisolid tion electrolyte is common for both PO2 and Pco2. The electrodes are dip coated with thin polystyrene diffusion membrane. When device is placed in blood, water vapour diffuses through the membrane and together with NHCO3 and NACL crystal deposited in hydrogel film constitutes the electrolyte normally used with PCO2 electrode. Under these conditions output signal from PO2 and PCO₂ is obtained. Describe the working thermal conductivity analyzer using thermistor with c) 04 neat diagram. 1. In thermal conductivity analyzer by using thermister, two thermistors are Ans. employed as heat sensing elements and two platinum filaments are used 02 in four arms of Wheatstone bridge.



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	2. Thermistor possesses the advantage of extremely sensitive to relatively	marks	
	minute change in temperature.	for	
	3. When used they are encapsulated in glass.	explanat	
	4. They are arranged in a constant current bridge circuit and each of them is	ion	
	placed in a separate cavity in a brass or stainless steel block (thermister is		
	encapsulated in glass).		
	5. The block acts as a heat sink. The material used for construction of		
	filaments must have a high temperature-coefficient of resistance.		
	6. The materials generally used for the purpose include tungsten, Kovar		
	(alloy of co, Ni and Fe) or platinum.		
	7. Two filaments connected in opposite arms of the Wheatstone bridge act		
	as reference arms, whereas the other two thermisters are connected in the		
	gas stream, which act as measuring arms. 8. The use of a four-cell arrangement serves to compensate for temperature		
	and power supply variations.		
	9. Initially, reference gas is made to flow through all the cells and the		
	bridge is balanced precisely with the help of potentiometer D.		
	10. When the gas stream passes through the measuring pair of thermister,		
	there is a corresponding change in the resistance of the thermister.		
	11. The higher the thermal conductivity of the gas, the lower would be the		
	resistance of the wire and vice versa.		
	Rs T1 (2) T2		
		02	
	$\S R_3$	Marks	
	Unbalance	for	
	H ₁ H ₂ current	Diagra	
	T indicator	m	
		111	
	Recorder Recorder		
	Tiscoide!		
	P		
	*		
d)	List any four applications of NMR	04	
Ans.	Applications of NMR:	01 mark	
	1) NMR is used in magnetic resonance imaging in medical diagnosis	for each	
	2) By studying peak of nuclear magnetic spectra, chemist can determine	applicat	
	structure of chemical compound.	ion (any	
	3) NMR is extremely used for analysis of sample non-destructively.	four)	
	4) NMR is used for data acquisition in petroleum industry and natural gas		
	exploration and recovery.		
			1
	5) NMR is used in process control and process optimization in oil refineries and petrochemical plant.		



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	6) It is a complex system integrating several technologies.		
	OR		
	Any other four relevant applications shall be considered		
04	Attempt any ONE		06
B)			
a)	Describe with neat diagram gas chromatography technique for the	06	
<u> </u>	measurement of Carbon Monoxide.	0.4	
Ans.	 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. The methane would pass through the reducing chamber unaffected while CO is reduced to methane. By using hydrogen flame ionization detector both methane peaks can be detected. The first peak is due to methane while the second peak would correspond to CO. The accuracy is about +-2%.peak heights of CO and CH4 would gives sensitivity of 50 ppb. 	04 Mark for explanat ion	
	Sample injector Pressure regulator Flow regulator Carrier gas Column	02 marks for diagram	
	Block Diagram of Gas Chromatograph		
	OR		
	(Any other relevant diagram shall be considered)		
b)	Describe the constructional details of flame photometer with neat diagram	06	
Ans.			

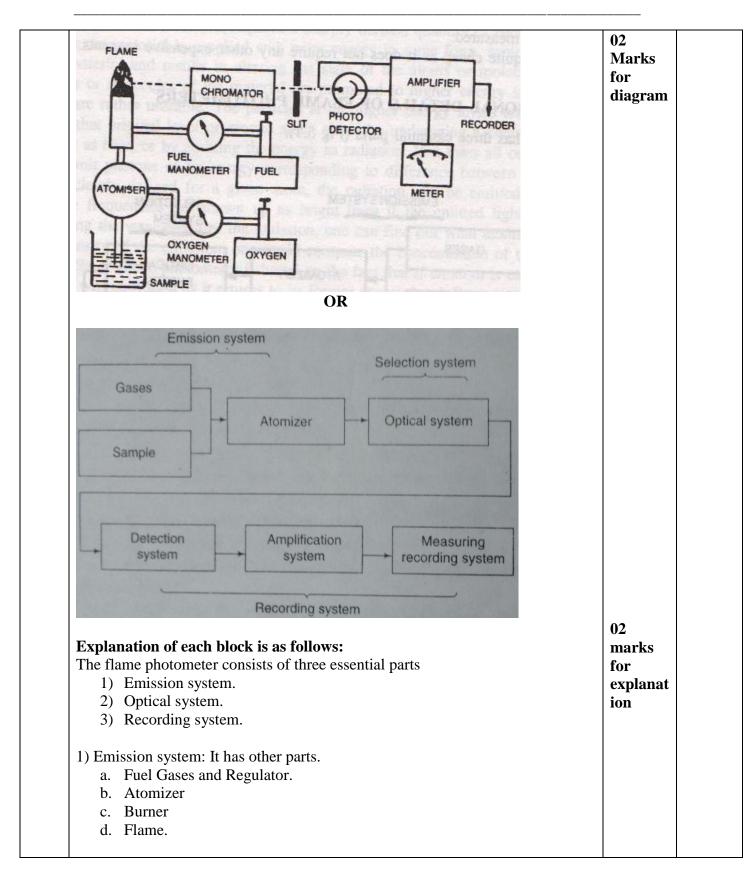


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a) Fuel Gases and their Regulation Pressure Regulators: In order to obtain a steady emission reading, it is needed to have steady flame. In order to achieve this, the air or oxygen and fuel pressure has to be maintained constant during the operation of the instrument. Suitable pressure gauges are therefore provided in the instrument to indicate the pressure that is actually present in the line. The fuel gas normally used in flame photometry is acetylene gas, which is commercially available in cylinders of various sizes. b) Atomizer: It is a system used to form aerosol (drop of liquid) by breaking atoms of liquid into small drops. This little device is responsible for introducing the liquid sample into the flame at a stable and reproducible rate. The atomizer must not be attacked by corrosive solutions. c) Burner: The burner brings the fuel, oxidant and sample aerosol together so that they may react safely and produce a good flame. d) Flames: It forms the source in which the light radiations characteristics of sample elements are produced. 2) Optical system: the emitted light by flame is passed through filters, monochromators and focussed on photo detector. This detector converts light energy into electrical signal. 3) Recording system: electrical signals o/p of detector is recorded or indicated using suitable photometer recorders using amplifier.		
Attempt one FOLD		16
State principle of calorimetric method. Describe working of double beam filter photometer with suitable diagram.	04	16
Principle of calorimetric method: Colorimetric method involves the measurement of colour in the visible region of electromagnetic spectrum. Colour measurement involves the absorption of visible light, which depends upon the concentration of a substance. Since colour of light is absorbed or transmitted depends on properties of the solution including the concentration of particles in it. Diagram:	01 Mark	
	Pressure Regulators: In order to obtain a steady emission reading, it is needed to have steady flame. In order to achieve this, the air or oxygen and fuel pressure has to be maintained constant during the operation of the instrument. Suitable pressure gauges are therefore provided in the instrument to indicate the pressure that is actually present in the line. The fuel gas normally used in flame photometry is acetylene gas, which is commercially available in cylinders of various sizes. b) Atomizer: It is a system used to form aerosol (drop of liquid) by breaking atoms of liquid into small drops. This little device is responsible for introducing the liquid sample into the flame at a stable and reproducible rate. The atomizer must not be attacked by corrosive solutions. c) Burner: The burner brings the fuel, oxidant and sample aerosol together so that they may react safely and produce a good flame. d) Flames: It forms the source in which the light radiations characteristics of sample elements are produced. 2) Optical system: the emitted light by flame is passed through filters, monochromators and focussed on photo detector. This detector converts light energy into electrical signal. 3) Recording system: electrical signals o/p of detector is recorded or indicated using suitable photometer recorders using amplifier. Attempt any FOUR State principle of calorimetric method. Describe working of double beam filter photometer with suitable diagram. Principle of calorimetric method: Colorimetric method involves the measurement of colour in the visible region of electromagnetic spectrum. Colour measurement involves the absorption of visible light, which depends upon the concentration of a substance. Since colour of light is absorbed or transmitted depends on properties of the solution including the concentration of particles in it.	Pressure Regulators: In order to obtain a steady emission reading, it is needed to have steady flame. In order to achieve this, the air or oxygen and fuel pressure has to be maintained constant during the operation of the instrument. Suitable pressure gauges are therefore provided in the instrument to indicate the pressure that is actually present in the line. The fuel gas normally used in flame photometry is acetylene gas, which is commercially available in cylinders of various sizes. b) Atomizer: It is a system used to form aerosol (drop of liquid) by breaking atoms of liquid into small drops. This little device is responsible for introducing the liquid sample into the flame at a stable and reproducible rate. The atomizer must not be attacked by corrosive solutions. c) Burner: The burner brings the fuel, oxidant and sample aerosol together so that they may react safely and produce a good flame. d) Flames: It forms the source in which the light radiations characteristics of sample elements are produced. 2) Optical system: the emitted light by flame is passed through filters, monochromators and focussed on photo detector. This detector converts light energy into electrical signal. 3) Recording system: electrical signals o/p of detector is recorded or indicated using suitable photometer recorders using amplifier. Attempt any FOUR State principle of calorimetric method. Describe working of double beam filter photometer with suitable diagram. Principle of calorimetric method: Colorimetric method involves the measurement of colour in the visible region of electromagnetic spectrum. Colour measurement involves the absorption of visible light, which depends upon the concentration of a substance. Since colour of light is absorbed or transmitted depends on properties of the solution including the concentration of particles in it.



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Model Answer

Cuvette Filter Measuring Mirror 01 photocell Mark diagram Intensity control Reference photocell 2 Working: The light from the lamp source is made to pass through the filter F and then is divided into two parts. One part will pass through the sample solution in cuvette and then fall on the measuring photocell. The part of light directly falls on the 02 reference photocell. Marks Galvanometer G which is kept in between the photocells receives opposing Workin currents through it. Potentiometer P₁is graduated in transmittance and absobance g limits. i) With lamp off,the galvanometer zero is adjusted mechanically. The potentiometer R_1 is set to T=1<u>OR</u>A=0ii) Then with the lamp on the blank solution is placed in the light path of the measuring cell. iii)Potentiometer P₂ is adjusted until galvanometer G reads to zero. iv)The solution to be analyzed is then substituted for the blank and P₁is adjusted until the current through galvanometer is zero, with setting R₂ remaining unchanged. v)The absorbance or transmittance can then be read directly on the scale of potentiometerP_{1.} Explain construction & working of null detector type pH meter. 04 b)



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Ans.	Diagram of null-detector type pH meter: $\begin{array}{c c} R_{\text{measurement electrode}} \\ \text{voltage} \\ \text{produced by} \\ \hline \\ R_{\text{reference electrode}} \\ \approx 3 \text{ k}\Omega \end{array}$ precision voltmeter	02 Marks for diagram
	Any other relevant diagram shall be considered	
	 Working of null-detector type pH meter: The null-detector type pH meter is uses a potentiometric 'null-balance' method. Fig shows the principle of this method in which the voltage output between a pair of pH electrodes is measured without drawing any current from the circuit under test. In the circuit, the emf developed on the pH electrodes is shown along with series resistors of both the glass electrode and reference electrode. The Precision voltage can be adjusted until the null detector shows zero. The reading on the voltmeter connected in parallel with the precision voltage would show the electrode potential representing pH of the solution. At the 'null' condition, there would be zero current in the pHelectrode circuit and therefore no voltage drop across the resistances of either electrode, giving the real electrode voltage at the voltmeter terminals. The pH value is read from the calibrated precision voltage source dial marked in pH units 	02 Marks for Workin g
c)	How measurement of ozone is done with the help of conductivity meter.	04
Ans.	Diagram:	02 Marks for Diagra m

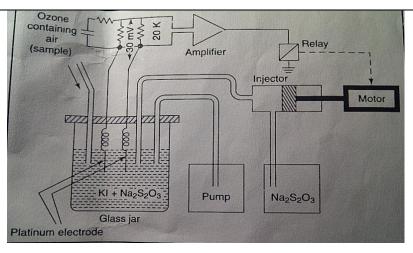


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02 Marks for Workin

Working:

A wet chemical method which uses the oxidizing properties of O_3 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 O_3 levels by previous calibration with known ozone source. Thus construct air-ozone meter which measures and records instantaneous 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 thiosulphate 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

$$O_3+2I+H_2O \rightarrow I_2+O_2+2OH^-$$

The iodine then reacts with thiosuphate

$$I_2+2(S_2O_3) \rightarrow 2(I-)+S_4O_6-$$

Reaction continues so long as there is thiosulphate in the solution. When all the thiosulphate 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



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	series with the electrode is used to operate the recorder as well as relaywhich 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 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.	
d)	Draw & describe circuit diagram for computation of total CO ₂ for blood gas analyzer.	04
Ans.	i) Blood gas analyzers are designed to measure pH, pCO ₂ , and pO ₂ from single sample of blood. ii) The input signal to HCO ₃ calculator comes from the outputs of pH amplifier and pCO ₂ amplifier. The outputs are suitably adjusted and given to the adder. The next stage is an antilog generator. iii) The total CO ₂ (TCO ₂) is calculated by summing the output signals of HCO ₃ and antilog output of pCO ₂ amplifier. iv) The output of this circuit goes to ADC for display.	02 Marks for Diagra m 02 Marks for Descript ion
e)	Define Chemiluminescense. How measurement of nitrogen oxide is done using Chemiluminescense.	04
Ans.	Definition: 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	01 Mark for Definiti on



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	possess higher energy levels than the ground state and usually have a very short life. Nitrogen oxide measurement using Chemiluminescence: i)Chemiluminescence phenomenon is very useful for measurement of air pollutants, particularly NO and NO ₂ . ii)Instruments based on the measurement of chemiluminescent emission, based on the following reaction have been developed: NO + O ₃ →NO ₂ + O ₂ NO ₂ → NO ₂ + hυ (λ max =6300 Å) iii)Since NO2reacts only slowly with ozone and the reaction which produces NO3 is not accompanied by Chemiluminescence, it is necessary to reduce to NO2 to NO beforeadmission into the reactor NO ₂ →NO + ½ O ₂ iv)Nitric oxide and ozone containing gas steam are mixed in a vessel at a sub atmospheric pressure of about 2 mm of Hg. v)Light emission is measured with a photomultiplier vi)With the use of high gain, low dark current photomultiplier tubes, extremely low levels of radiation can be measured. vii)The response of the instruments based on Chemiluminescence is linear from 1 ppb to 1000ppm of NO. viii)This technique is extremely useful for measurement of NO in automotive exhaust gases.	03 Marks Descript ion	
f)	List any four applications of Chromatography.	04	
Ans.	Applications of Chromatography i)Biochemical Screening for Genetic Disorders ii)Separation of nonvolatile and thermally unstable samples as well as polymers and inorganic salts.	01 Mark for each applicat ion	



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	 iii)Ability to both qualitatively and quantitatively analyzes multicomponent streams with the versatility to analyze a wide range of samples. iv)Analysis of biological fluids, v)Therapeutic Drug Monitoring and Toxicology, vi)R&D in pharmaceutical industries, vii)Vitamins and Related Metabolites, viii)Steroid Hormones 		
06			16
06 a)	Attempt any FOUR List any two applications of each	04	16
	i)GCMS ii)LCMS		
Ans.	Application of GCMS & LCMS i)GCMS applications:	01	
	 Environmental monitoring and clean up 	Mark	
	Criminal forensic	for each	
	Sports antidoping	(Any	
	• Explosion detector	two)	
	 Air port security for detecting luggage or items in human body. 		
	 ii)LCMS applications: Bio analysis. Meta bolamics – to detect over thousands of proteins from body Drug research , production Analysis of organic or inorganic products. 	01 Mark for each (Any two)	
b)	What is electrophoresis? List part of electrophoresis apparatus.	04	
Ans.	Electrophoresis: 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	03 Marks for explanat ion	



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	negatively. Ions have different migration rates depending upon their total charge, size and shape, and can therefore be separated.		
	The moving boundary method of electrophoresis utilizes the migration of the particles in free solution and observation of various molecular boundaries through sensitive refracts metric techniques. With this the value of electrophoresis in obtaining distinct and measurable fractions of variety of substances got well established particular in clinical laboratories. 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 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.		
	The parts of electrophoresis apparatus: 1)Electrophoresis cabinet 1.1. Plastic cabinet 1.2. Gable cover 1.3. Carrier rack 2)Power supply 3)Densitometer	01 Mark for list	
c)	Draw optical diagram of spectrometers using prism. State role of prism in it.	04	
Ans.			



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Model Answer

Diagram: Prism movement Pivot 02 **Marks** Prism arm Aluminized for surface diagram Sample compartment Beam attenuator Condensing lens Fxit slit Light source **Entrance** slit Photocell Role of prism: 1) Light is made to fall on the prism. The monochromatic light is obtained by 02 allowing the light beam to pass through a prism monochromator. Marks 2) Prism is an optical component to disperse the light or modify the direction of for light. explanat 3) The wavelength selection is done by rotating the prism about a pivot. ion 4)The prism has an aluminized rear surface. Due to the prism, shorter wavelength is dispersed more. Light, after getting dispersed by prism, passes through the sample and then detected by the photocell. 04 d) Describe with neat diagram Time of flight mass spectrometer. 02 Diagram of time of flight mass spectrometer Ans. Marks for Control grid (pulsed) Potential diagram Filament selector Electron beam Sample Ion beam inlet Oscilloscope **Dynodes** Magnetic Ion Accelerating electron source region multiplier Anode Drift path Ion signal collector and amplifier



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	Working:		
	i)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.		
	ii)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.	02 Marks	
	iii) In pulse mass spectrometer, ion packets of a few microseconds duration are emitted at intervals of a few milliseconds from a voltage source.	for explanat	
	iv)The ions transverse an evacuated tube called the drift tube to reach the detector.	ion	
	v)The detector is sensitized for a brief instant to register their arrival.		
	vi)Since ions of different masses arrive at the detector at different times, the accurate measurement of the time between activating the source and sensitizing the detector gives information concerning the mass of the ions.		
	$\frac{m}{s} = \left(\frac{2V}{L^2}\right) * t^2$		
	Mass/charge ratio is directly proportional to time taken		
	vii)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 theoscilloscope commences as the ion packet start out.		
	viii)This produces a mass spectrum on the screen of the oscilloscope.		
e)	Give classification of chromatography. Also enlist different detection system used in chromatography.	04	



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Ans.	Classification of Chromatography. Chromatography. Chromatography. Liquid Chromatography. Gas Iliquid. Gas Isolid. paper Column. Thin layer. (partition) (adsorption) Liquid/Liquid. Liquid/solid. Gel. Ion (partition) (adsorption) permeation Exchange	02 Marks for classific ation
	The various detectors used in gas chromatography are: • The katharometer or thermal conductivity detector • Flame ionization detector • Flame photometric detector • Photoionization detector • Electron capture detector • Argon ionization detector • Cross section ionization detector • Atomic emission detector • Chemiluminiscence spectroscopy-based detector • Nitrogen-phosphorus detector	02 Marks for listing