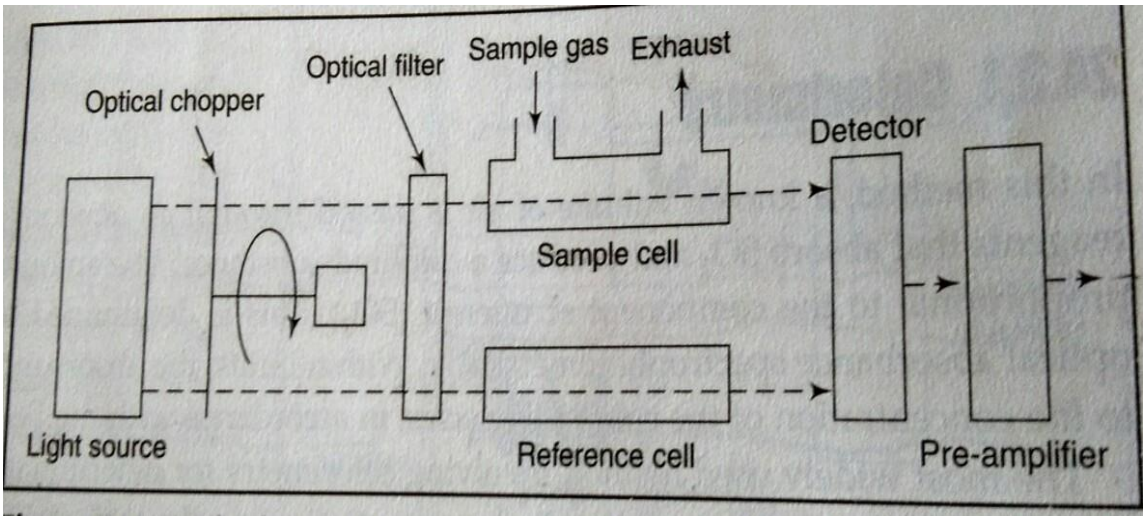
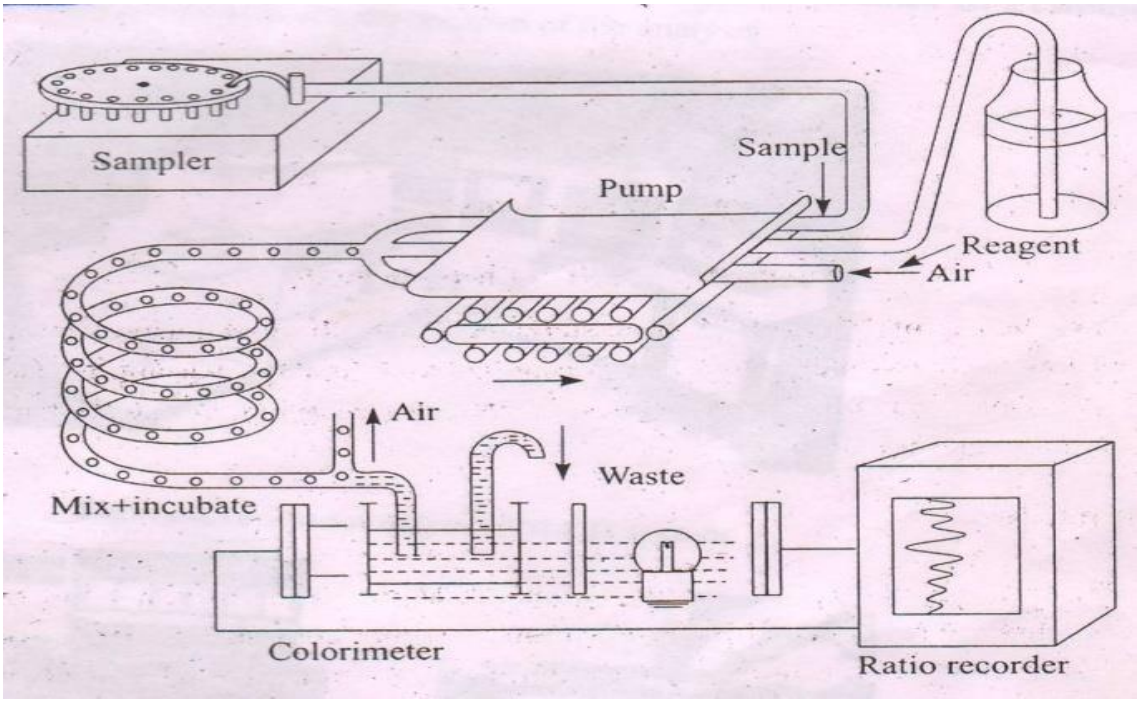
SUMMER – 2019 EXAMINATION

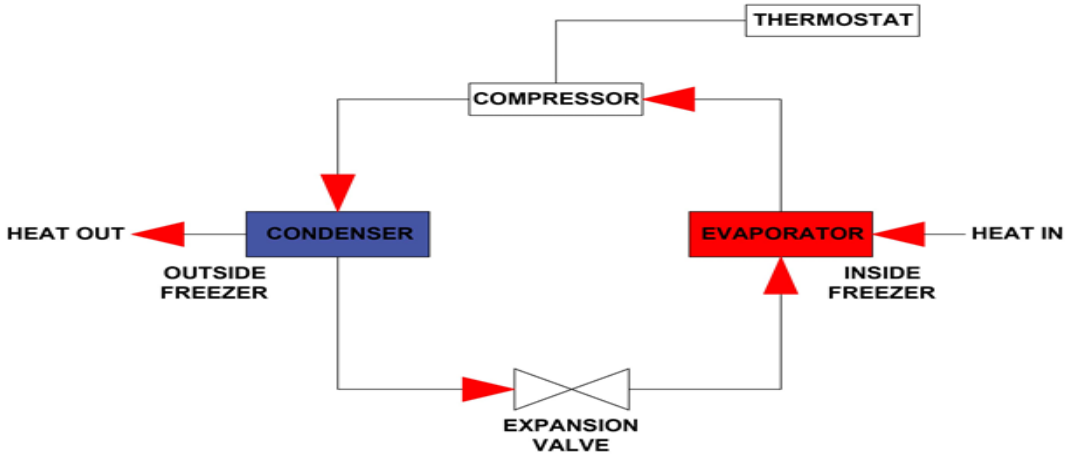
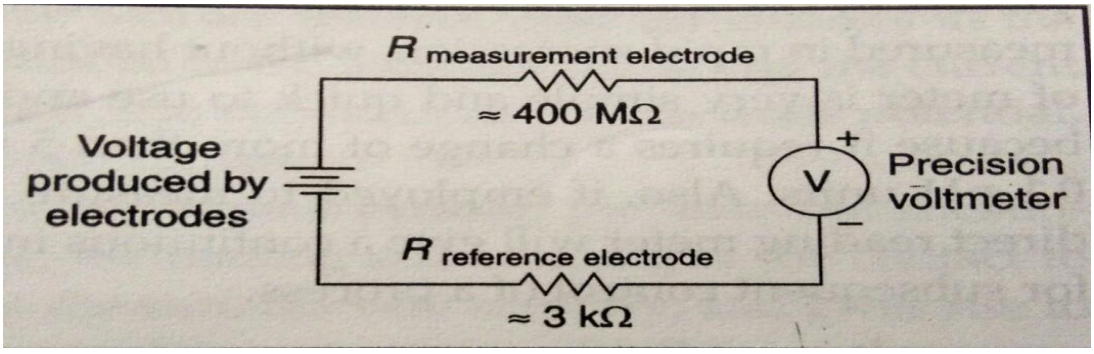
Subject Name: Analytical Equipment **Model Answer** **Subject Code:** 22435

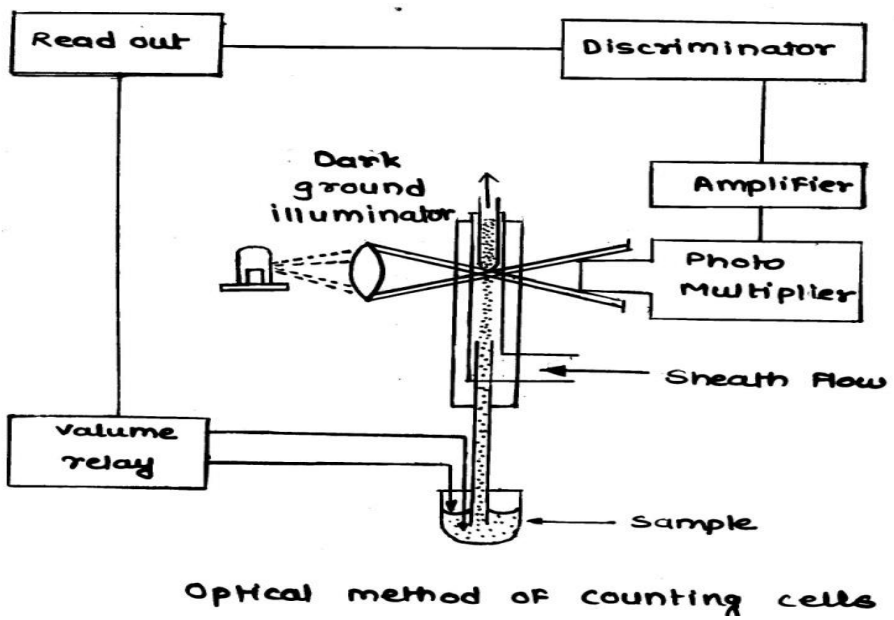
Important Instructions to Examiners:

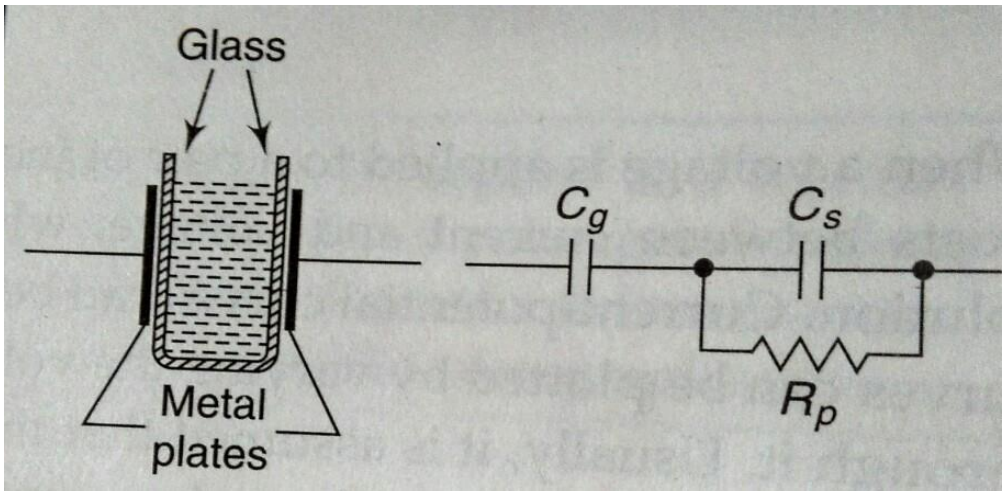
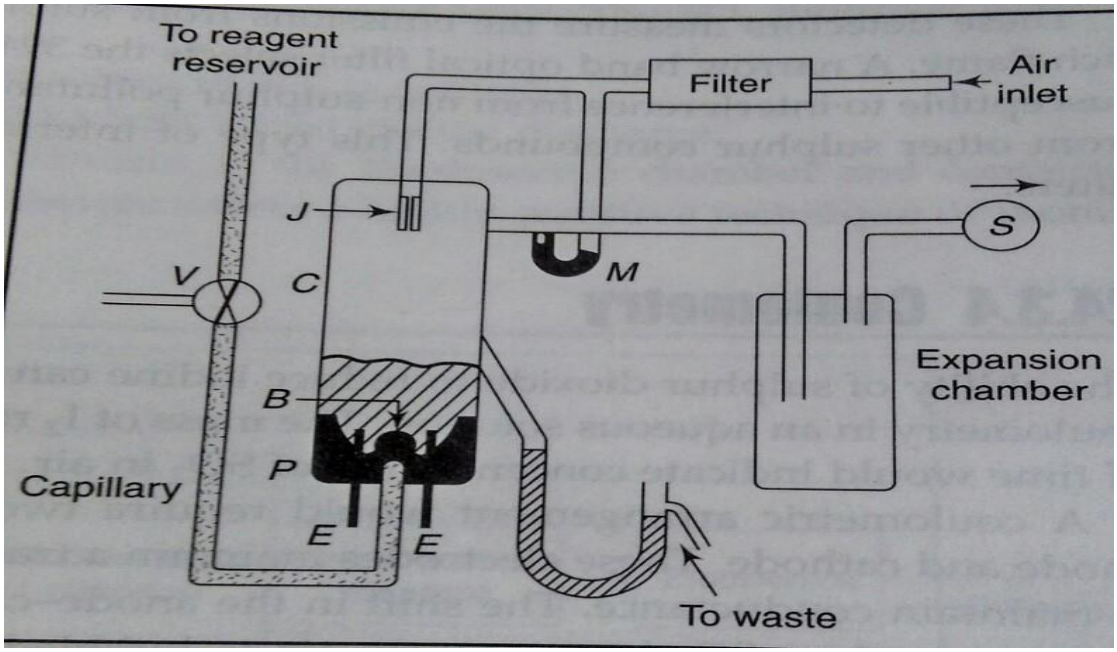
1) The answers should be examined by key words and not as word-to-word as given in the model answer scheme.
2) The model answer and the answer written by candidate may vary but the examiner may try to assess the understanding level of the candidate.
3) The language errors such as grammatical, spelling 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.

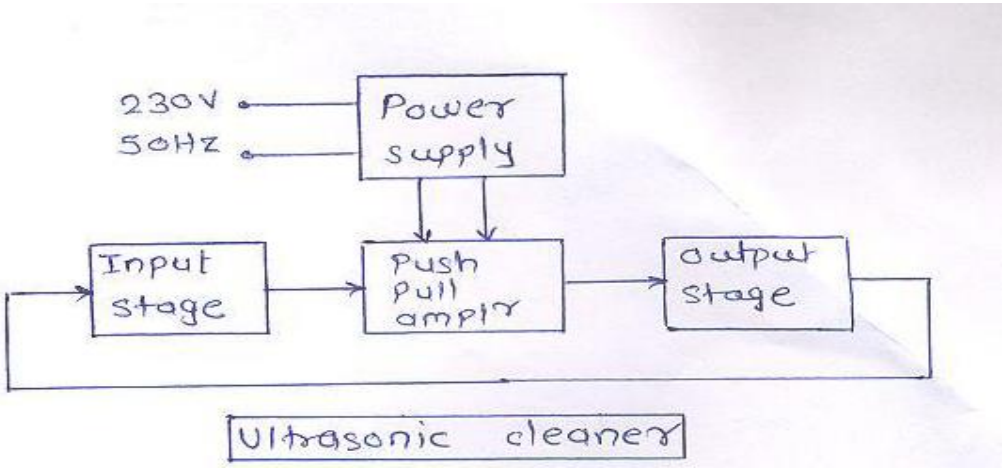
Q. No.	Sub Q. N.	Answer	Marking Scheme
1.		**Attempt any FIVE of the following:**	**10 M**
	(a)	**List any two applications of flame photometer.** **Ans:** **Applications of flame photometer:** 1. Determine the concentrations of Na+ and K+ ions are present in the human body fluids. 2. Determine the presence of various alkali and alkaline earth metals in soil sample. 3. Determine the hardness of water. 4. Determine lead present in the petrol. 5. The amount of metals present in the waste water.	**02M**
	(b)	**List any four applications of incinerator.** **Ans:** **Applications of incinerator:** This is mostly used for the purpose of burning biomedical waste like 1. Burning of Placenta 2. Disposable needle syringes 3. Surgical pads 4. Hand glows etc which are used in hospital 5. To burn hygienic waste generated daily may be also saline bottles, dressing cottons & dangerous body parts. etc	**02M**
	(c)	**Define electrophoresis.** **Ans:** **Definition of electrophoresis:** Electrophoresis is a method of analytical chemistry. Basically electrophoresis technique is to separate the molecules based on charge under the influence of an electric field.	**02M**
	(d)	**Suggest the meter to measure hydrogen ion concentration in the given solution.** **Ans:** **Measure hydrogen ion concentration:** The pH meter is used.	**02M**

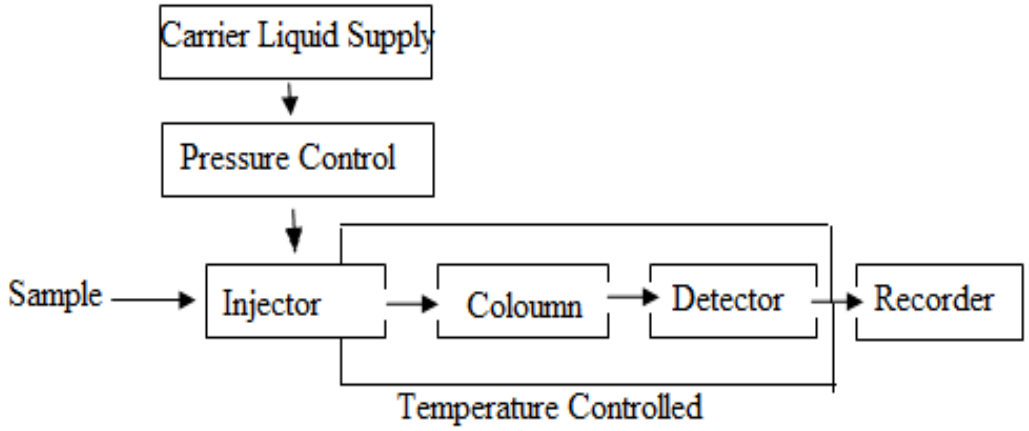
	(e)	**Draw a neat labelled diagram of non- dispersive infrared analyzer for carbon monoxide measurement.** **Ans:**  **Fig: Non-dispersive infrared analyzer for carbon monoxide measurement**	**02M**
	(f)	**Suggest suitable sterilizing methods for sterilization of medical devices.** **Ans:** 1. Autoclave 2. Ultrasonic cleaner	**01M** **01M**
	(g)	**Write the effect of temperature on pH measurement.** **Ans:** The pH decreases with increase in temperature. But this does not mean that water becomes more acidic at higher temperatures. A solution is considered as acidic if there is an excess of hydrogen ions over hydroxide ions.	**02M**
2.		**Attempt any <u>THREE</u> of the following:**	**12 M**
	(a)	**Draw block diagram of auto - analyzer and describe function of each block.** **Ans:**  **Fig: auto - analyzer**	**02M**

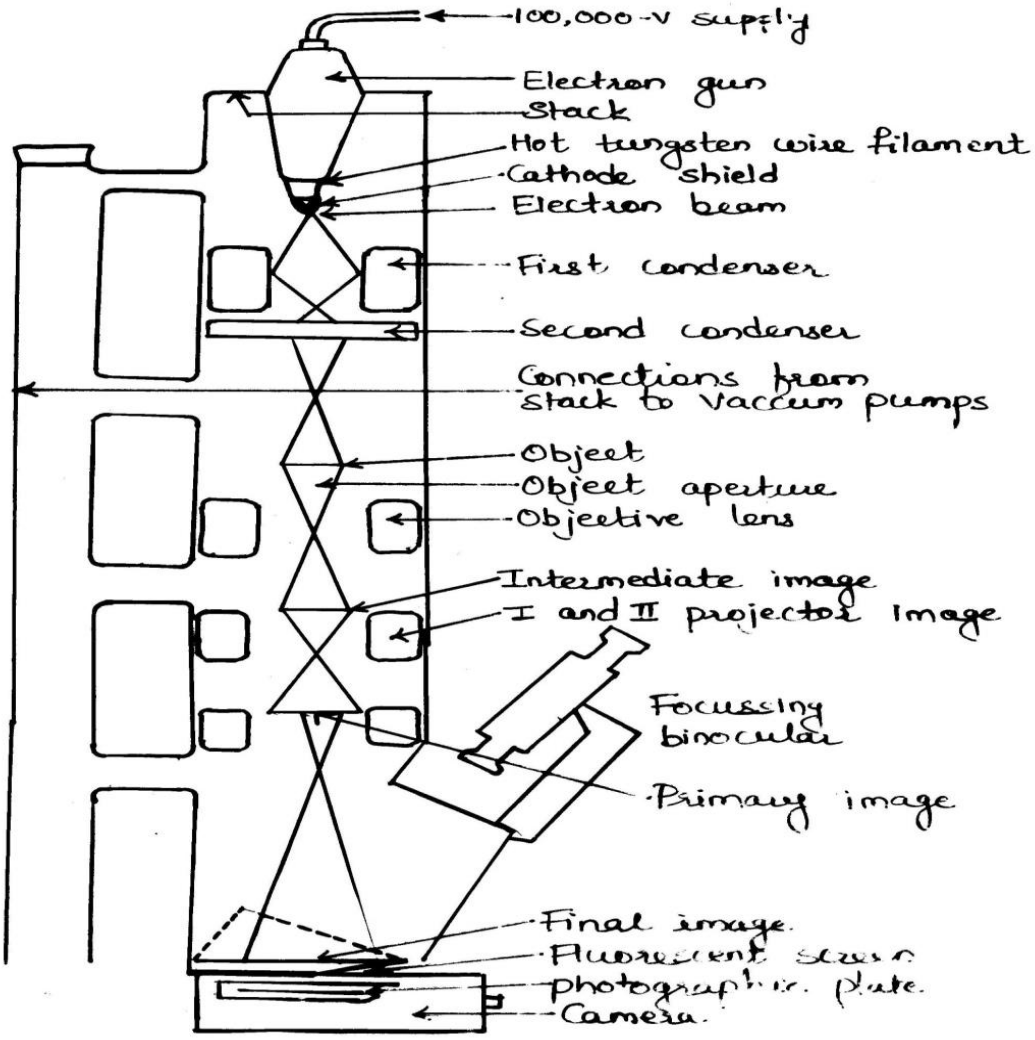
		Sampler: It fits the sample into analyzer in a particular time sequence. **Proportionality pump:** It is used to mix the sample & regent. **Mixing:** Mixing is achieved by injecting air bubbles and the mixture is incubated or heated while flowing through heated coils. The air bubbles are removed & the solution finally passes toward the colorimeter or is aspired in to flame photometer. **Colorimeter:** It monitors the changes in optical density of the fluid stream flowing through a tabular flow cell. Color intensities (optical densities) proportional to substance concentrations are converted into the equivalent electrical voltages. **Recorder:** It is used to convert electrical signal from the colorimeter into a graphic display on a moving chart.	**02M**
	(b)	**Describe freezer with a neat labelled block diagram.** **Ans:**  **Fig: Block diagram of freezer** The compressor constricts the refrigerant vapour, raising its pressure and temperature, and pushes it into the coils of the condenser on the outside of the refrigerator. When the hot gas in the coils of the condenser meets the cooler air temperature of the kitchen, it becomes a liquid. Now in liquid form at high pressure, the refrigerant cools down as it flows through the expansion valve into the evaporator coils inside the freezer and the fridge. The refrigerant absorbs the heat inside the fridge when it flows through the evaporator coils, cooling down the air inside the fridge. Last, the refrigerant evaporates to a gas due to raised temperature, and then flows back to the compressor, where the cycle starts all over again. The main component of a refrigerator that needs power is the compressor. It is essentially a pump which is driven by a motor. The thermostat controls the temperature of the fridge by switching on-and-off the compressor.	**02M** **02M**
	(c)	**Draw neat circuit for null detector type pH meter.** **Ans:**  **Fig: Null detector type pH meter**	**04M**

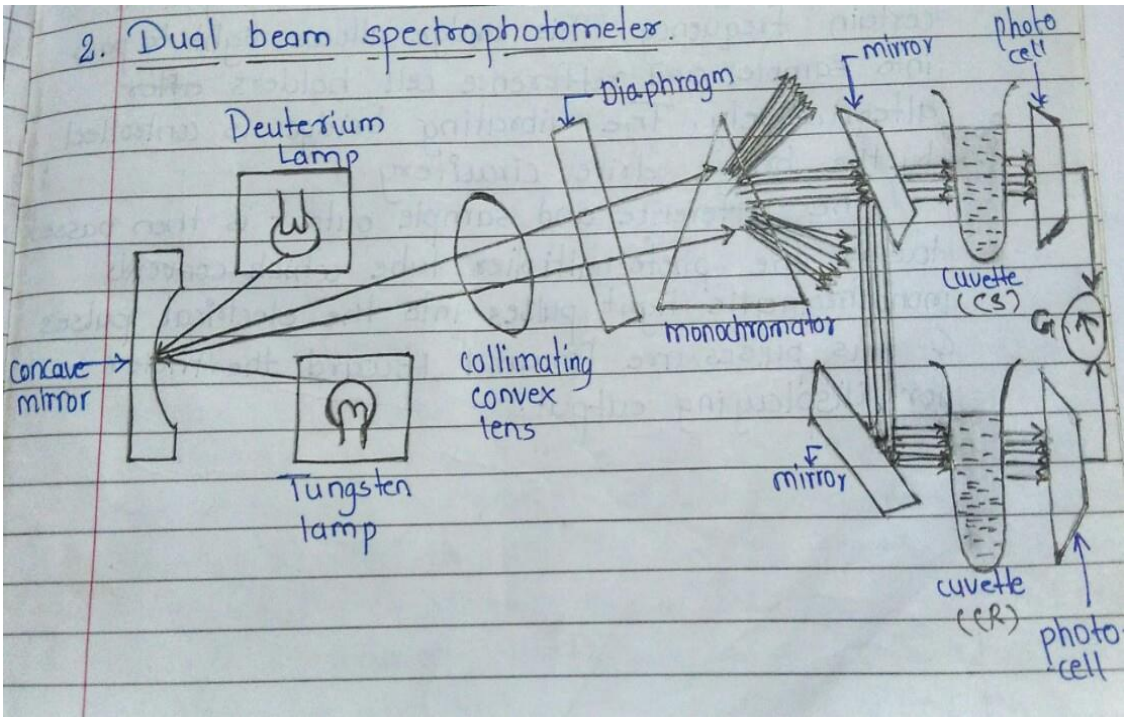
	(d)	**Following are the full scale measurement range for various gas pollutants. Suggest measurement technique for following pollutants:** **i. Nitrogen oxides: 0 - 1 ppm** **ii. Oxidants: 0 - 500 ppb** **iii. Sulphur oxide: 0 - 2 ppm** **Ans:** **Nitrogen oxide (0 - 1 ppm):** Colorimetric method, Coulometric method, Chemiluminescence method, Electrochemical transducers, Infrared spectroscopy, Lasers. **Oxidants (0 - 500 ppb):** Chemiluminescence method, Coulometric method, Colorimetric method, Ultraviolet absorption method. **Sulphur oxide (0 - 2 ppm):** Ultraviolet absorption, Infrared absorption, Gas chromatography (Flame photometric detector), Colorimetric method, Conductimetric method, Coulometric method, Electrochemical transducers.	**04M**
3.		**Attempt any <u>THREE</u> of the following:**	**12 M**
	(a)	**Suggest sterilizing equipment used for following application:** **i. For various powders in medical use.** **ii. For sterilizing biomedical waste.** **Ans:** **For various powders in medical use:** Hot air oven **For sterilizing biomedical waste:** Autoclave	**02M** **02M**
	(b)	**Describe working principle of dark field blood cell counter with neat diagram.** **Ans:**  **Fig: Dark field blood cell counters** Optical method used to counting blood cells. The method is based on collecting scattered light from blood cells & converting into electrical pulses for counting of red & white cells using optical detection system The sample of dilute blood 1:5000 for white cells 1:5000 for red cells is taken in a glass container it is drawn through counting chamber in which the blood stream is reduced in cross section by a concentrated high velocity liquid sheath. A sample optical system provides a dark field illumination & the light scattered in forward direction is collected on the cathode of a photomultiplier tube. Pulses are produced through PMT corresponding to each cell. These cells are amplified in a high input impedance amplifier & fed to an adjustable amplitude discriminator. The	**02M** **02M**

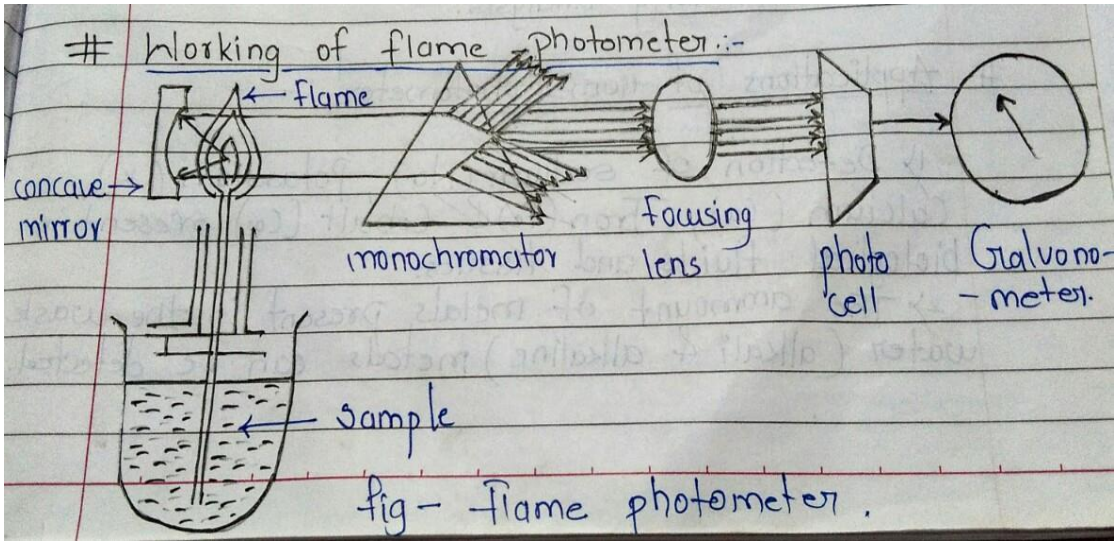
		discriminator provides pulses of equal amplitude which are used to drive a digital display. Instruments based on this technique takes about 30 seconds for completing the count the accuracy of 2% is attainable, the instrument required about 1mm of blood sample.	
	(c)	**Draw equivalent circuit diagram of conductivity cell and describe it.** **Ans:**  **Fig: Equivalent circuit of conductivity cell** A typical sample cell for high frequency conductivity measurement may be composed of two metallic plates sealed on to the wall of a rectangular container. When the solution is put in the container, the metal plates act as a condenser with solution and glass as the dielectric. The equivalent circuit of the conductivity cell is shown in fig. C_g represents the capacitance of the glass walls of the cell, C_s is the capacitance of the sample and R_p is the resistance in parallel with C_s. The resistive component is very high and offers negligible contribution. Capacitive effect is the major factor in high frequency measurements, whereas resistive balance is more important in low frequency measurements.	**02M** **02M**
	(d)	**Describe conductivity method with neat diagram for measurement of SO_2 in air.** **Ans:**  **Fig: Conductivity method for measurement of SO_2 in air**	**02M**

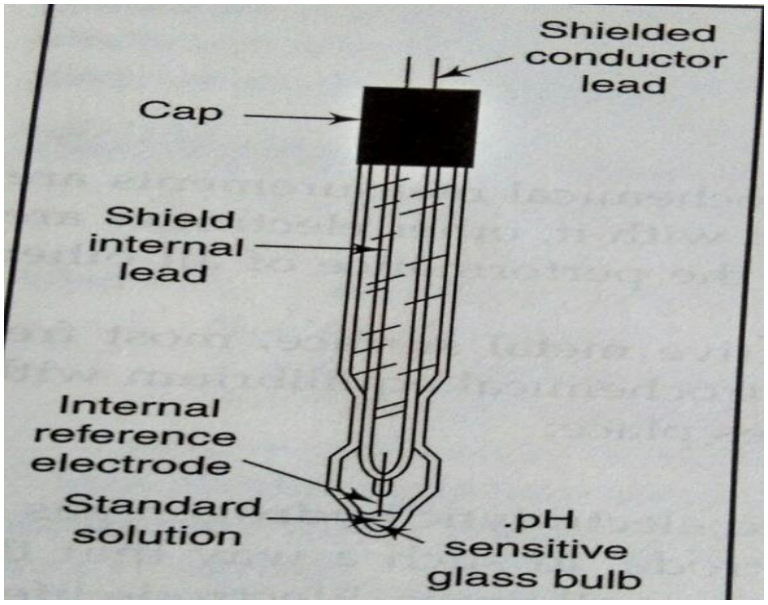
		Conductivity cell is used for the continuous measurement of Sulphur dioxide in air. The cell C is made of glass, 1 cm inside diameter with a jet J, orifice 0.5 mm diameter and located 1 cm above the reagent surface. Two electrodes E made of 18 SWG stainless steel wire are inserted through a Perspex cap P. The cap is sealed to the base of the cell. Reagent enters the cell from a central tube inserted in the Perspex cap. A small glass bead B in the cell acts as a non-return valve on the entry of the 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 materials. The flow of air through the jet is maintained at approximately 200 ml/min. The cell is of small size (1.5 ml) and its capacity to absorb SO_2 is thus limited. The electrolyte being discharged and replaced at regular intervals of 15 min. The resulting output is recorded as a saw tooth waveform. To measure the conductivity of the cell, 5v alternating current is applied across the electrodes. Alternating current avoids polarization. When normal urban concentrations of SO_2 are measured, the current through the cell increases from its zero value of 20µA to up to 40µA at the end of 15 min. sampling period. In conditions of heavy pollution, it may go to two milli ampere. Because the current is recorded every 15 minutes, the concentration of Sulphur dioxide at any instant is proportional to the slope of the saw tooth at that instant. The calibration is carried out by using known concentrations of Sulphur dioxide in air.	**02M**
4.		**Attempt any <u>THREE</u> of the following:**	**12 M**
	(a)	**Describe the steps for calibration of colorimeter.** **Ans:** **Calibration steps of colorimeter:** 1. Slide the lid of the colorimeter open to uncover the cuvette slot. 2. Insert a cuvette, filled with distilled water or other solvent used to prepare your solutions, for your calibration blank transmittance (100%) or absorbance (0). Slide the colorimeter lid closed. 3. Press the CAL button on the colorimeter to begin the calibration process. Release the CAL button when the red LED begins to flash. 4. When the red LED stops flashing, the calibration is complete. The absorbance reading should be very close to 0.000 (100%T). 5. Remove the blank cuvette from the Colorimeter.	**04M**
	(b)	**Explain ultrasonic cleaner with a neat diagram.** **Ans:**  **Fig: Ultrasonic cleaner**	**02M**

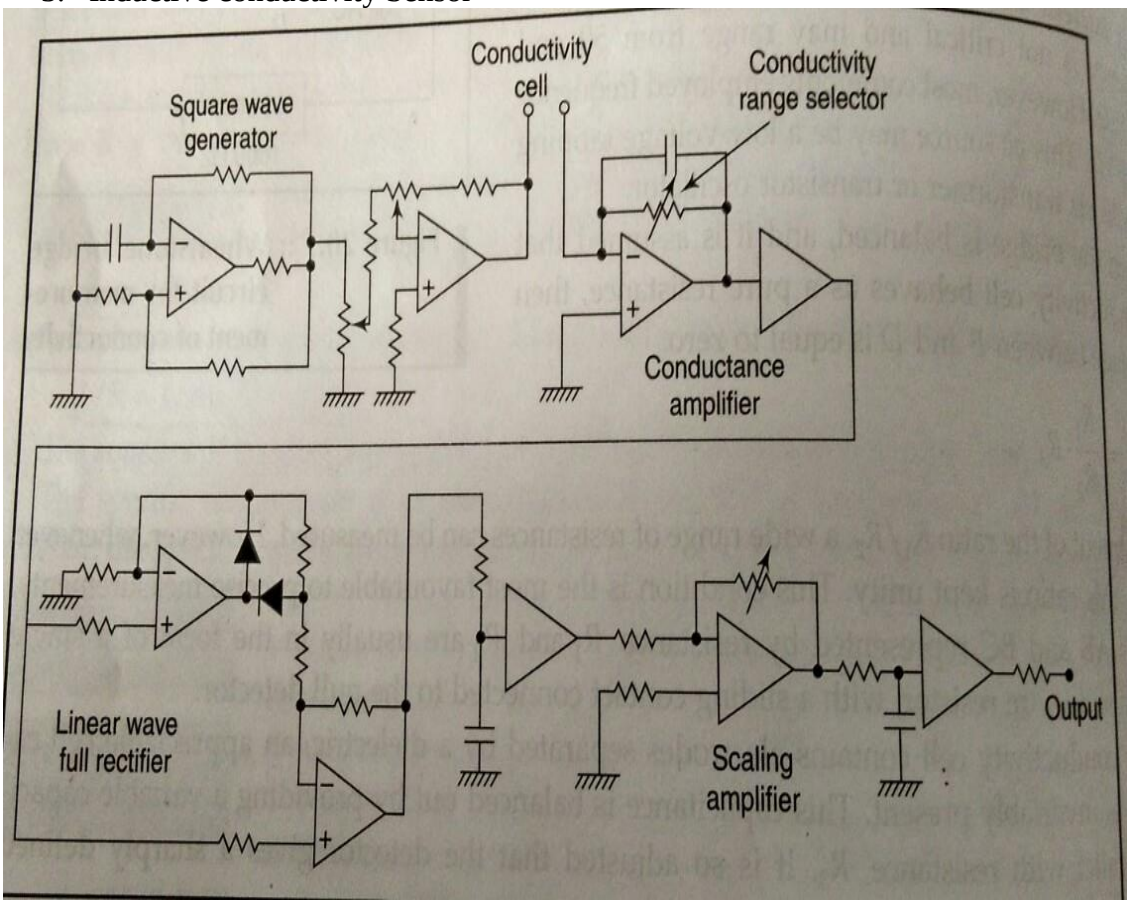
		Ultrasonic cleaner referred to as sonicator, is a cleaning device that uses ultrasound from (15 to 400 kHz) & appropriate cleaning solution to clean delicate items. The ultrasound is not effective without the cleaning solution. Enhance the effect of a solution for the item to be cleaned & the soiling. Ultrasonic cleaner produces high frequency waves through water & consequently cavitation's process cleans the instruments. The principle of ultrasonic cleaning process is as follows. Piezoelectric transducer is attached to cleaning tank. They generate ultrasonic waves that vibrate the cleaning fluid at very high velocity creating a process called calibration. Millions of tiny bubbles employed within solution can penetrate into every orifice of the item being cleaned removing dirt within seconds.	**02M**
	(c)	**Describe liquid chromatography with a neat labelled diagram.** **Ans:**  Fig: Liquid Chromatography The basic parts of a liquid chromatography are shown in figure. It consists of the following parts. 1. Carrier liquid supply along with pressure control. 2. Sample injection system. 3. Chromatographic column 4. Thermal compartment or thermostat 5. The detection system 6. The strip chart recorder Liquid Chromatography is an analytical chromatographic technique which is useful for separating ions or molecules that are dissolved in a solvent. Since these techniques used liquid as the percolating agent, liquid chromatography can be considered as the oldest of all chromatographic processes. Liquid chromatography consists of carrier liquid supply along with pressure control system used for transport the inserted sample to the system. Sample injection system is used to provide constant volume injection at sample into the carrier liquid. The solute mixes with the flowing carrier liquid and is passes toward the chromatographic column, which is the heart of the chromatograph. It is there that the different solutes in the vaporized sample are separated from each other. At the end of the column the solutes enter the detector which produces an electrical signal corresponding to the quantity of solute leaving the column. The detector signal is supplied to a recorder and a plot of the time signal amplitude called chromatogram is obtained. This record is used to determine the identity of the components in the mixture and their respective concentrations.	**02M** **02M**

	(d)	**Explain the working of transmission electron microscope with neat diagram.** **Ans:**  **Fig: Transmission Electron microscope (TEM)** The transmission electron microscope & its optical system are shown in fig: The electron microscope the top of which is mounted the source of illumination, the electron gun which emits electrons. A high voltage which can be varied from 50-100 kV is applied to the anode. A pencil beam of electron viewing on high velocity is projected through hole in the anode & onwards down the stack. The stack is completely evacuated & vacuum is maintained. Focusing & magnification are achieved by electromagnetic lenses. There is a condenser lenses system, which bends the rays of electron so that a parallel beam is directed on to the object placed below it. the electrons are scattered to a degree i.e. proportional to the thickness & density of the various parts of the specimen an objective lens gathers the scattered electrons through a very small & bring them to a focus were a real primary image is formed & is magnified about a 100 times two projector lenses, which have the functions the eye piece of the light microscope, magnify a part of primary image further 300 to 500 times. The focal length of the magnetic lenses can be changed by varying the current flowing through the lens & thus a continuously variable magnification is obtained. The final image is observed on a fluorescent screen situated at the lower end of stack & is viewed through a glass window. The screen can be withdrawn by to allow the electrons to impinge on a photographic plate or film held in a camera.	**02M** **02M**

	(e)	**Describe the concept of automated wet chemical air analysis.** **Ans:** Automated wet chemical air analysis in which the sample is automatically introduced and prepared. Reagents are then added in proper quantities sequence. A chemical reaction will take place. The presence of a particular constituent is then detected, displayed and recorded. In any air analysis system, the equipment must be capable of obtaining a proper sample. This is essential, in order to obtain correct results. In automated wet chemical methods, the accuracy of the sampling system is dependent upon maintaining constant ratio between the amounts of sample ratio and absorbing solution. The rate of flow of the absorbing solution is maintained constant by a proportionating pump.	**04M**
5.		**Attempt any <u>TWO</u> of the following:**	**12 M**
	(a)	**Describe dual beam spectrophotometer with neat diagram and list its applications.** **Ans:**  **Fig: Dual beam spectrophotometer** Dual beam spectrophotometer in which two lamps are used namely deuterium lamp and tungsten lamp, which is used for generate the radiations. These radiations forward toward the concave mirror and reflect radiations toward the collimating convex lens. This lens used for move radiations parallel toward the diaphragm and this is used for set 100 % transmission of the radiation. Then radiations passes toward the monochromator and it is used for selecting one wavelength of radiation. This radiation passes toward the two mirrors for reflecting radiations toward the sample solution and reference solution. Some of the radiations absorb the sample solution and remaining passes toward the detector (photocell) for converting radiations into the electrical signal. Finally output of detector is given to the galvanometer for display the output. **Applications of spectrophotometer:** 1. It is used to detect colour concentration of unknown sample. 2. It is used in laboratory for study of chemical substances present in sample. 3. It is used to determine the absorbance of the solution. 4. It is used for measurement of transmittance of the solution.	**02M** **02M** **02M**

	(b)	**Draw labelled diagram of a flame photometer. Describe its working.** **Ans:**  **Fig: Flame photometer** When the fuel gas is burned at the tip of the burner. The sample solution moves upward and then solvent is vaporized and solid molecules are produce. These solid molecules convert into the gas atoms. Some of the gas atoms absorb heat energy and jump into the excited state and instantaneous return to the ground state with emission of the radiation. These emitted radiations are collected by the concave mirror and are passing toward the monochromator. The monochromator is used for select one wavelength of radiation and forward toward the focusing lens. Focusing lens is used to collect the radiations coming from the monochromator and passes toward the detector (photocell). The detector is used for convert radiation into the electrical signal. Here, photocell used as a detector, then output of the photocell is given to the galvanometer for display the output.	**03M** **03M**
	(c)	**Describe the concept of PAGE in detail.** **Ans:** Electrophoretic techniques separate charged molecules in an electric field. The mobility of a molecule is inversely proportional to its size and directly proportional to its charge. During electrophoresis, proteins move towards an oppositely charged electrode in an electric field. The rate of their movement in an electrophoretic system is governed by several factors such as temperature, pH, and buffer concentration in addition to intrinsic properties such as the size, charge and shape of the proteins. Electrophoretic separation of proteins strictly on the basis of their molecular weight is possible only if the charge of all the protein molecules can be manipulated to the same sign. In such a case, the mobility of the protein molecules will be fully reliant on their size. Polyacrylamide gel electrophoresis (PAGE) is a technique based on this idea and is used to separate proteins on the basis of their size. In PAGE, an anionic detergent called sodium dodecyl sulfate (SDS) is used to bind to proteins and give them a negative charge. Proteins are then separated electrophoretically according to their size using a gel matrix made of polyacrylamide in an electric field. Polyacrylamide is produced as a result of the polymerization reaction between acrylamide and N, N'-methylene-bis-acrylamide (BIS) using a catalyst. The degree of polymerization or cross-linking can be controlled by adjusting the concentration of acrylamide and BIS. The more cross-linking harder the gel. Hardness of	**06M**

		the gel, in turn, modulates the friction experienced by macromolecules when they travel through the gel during PAGE, thus affecting the resolution of separation. Loose gels (4-8% acrylamide) allow higher molecular weight molecules to migrate faster through the gel while hard gels (12-20% acrylamide) restrict the migration of large molecules and selectively allow small ones to move through the gel.	
6.		**Attempt any <u>TWO</u> of the following:**	**12 M**
	(a)	**State two applications of the following analytical equipment:** **1. Auto-analyzer** **2. Centrifuge** **3. Autoclave** **Ans:** **Applications of auto analyzer:** 1. Auto analyzer determines levels of calcium, glucose, cholesterol, phosphate, proteins and uric acid in blood serum or other bodily samples. 2. Testing the chemical properties of various substances. 3. Test the composition of various compounds. 4. Soil testing laboratories 5. Fertilizer analysis 6. Sea water analysis 7. Tobacco leaf analysis **Applications of Centrifuge:** 1. Blood sample separation 2. DNA / RNA separation 3. Cell culture separation **Applications of autoclave:** 1. It is used to kill microorganisms and spores. 2. It is used to sterilize surgical equipment, laboratory instruments, pharmaceutical items, and other materials such as gowns, dressing and gloves. 3. Sterilization of waste materials.	**02M** **02M** **02M**
	(b)	**Describe construction details of glass electrode with neat diagram. List any four applications of pH meter.** **Ans:**  **Fig: Glass electrode**	**02M**

		The glass electrode consists of a thin walled bulb of pH sensitive glass sealed to a stream of non pH sensitive high resistance glass. The pH response is limited entirely to the area of the special glass membrane. , thus making the response independent of the depth of immersion. The membrane normally has a thickness of the order of 0.05 to 0.15 mm, and the bulbs are of the order of 10 mm in diameter. Fig. shows typical construction of a glass electrode. Both surfaces of the membrane are pH sensitive. **Applications of pH meter:** pH Meters are used, 1. To measure hydrogen-ion concentration (acidity or alkalinity) in solution. 2. Waste water treatment. 3. Pharmaceuticals. 4. Chemicals & petrochemicals. 5. Food & beverages. 6. Mining. 7. Water treatment plants.	**02M** **02M**
	(c)	**List conductivity sensors for measuring conductivity. Describe direct method for conductivity measurement with neat diagram.** **Ans:** **Conductivity sensors for measuring conductivity:** 1. 2-Electrode conductivity Sensor 2. 4-Electrode conductivity Sensor 3. Inductive conductivity Sensor  **Fig: Direct method for conductivity measurement** Direct reading instruments are preferred over the null balance type instruments. In these instruments, the necessity of converting resistance readings into conductance readings is eliminated. The unbalance bridge current is amplified in an	**02M** **02M**

		electronic amplifier and displayed on a calibrated panel meter. The excitation for the conductivity cell is a square wave, generated by a simple astable multivibrator constructed around an operational amplifier. Its frequency is adjusted to 10 kHz. The conductance amplifier is a wide band high gain operational amplifier, in which difference feedback resistors can be selected to obtain different conductance ranges between 0.2 and 10^{-7} Ω^{-1}. This is followed by a full wave rectifier for rectifying the output of the conductance amplifier. The output stage of the circuit is a scaling amplifier. The DC signal from the rectifier is in volts 0.5 * 10^{n} times the conductance of the solution, where n is a scale factor, introduced by the conductance amplifier feedback resistors. This is multiplied by 2 to obtain an output signal that is a simple factor of 10 of the conductance. The output of the amplifier is fed to the oscilloscope, via a logarithmic amplifier. The arrangement gives a straight line, with a slope equal to the rate constant of the reaction with reasonably good linearity.	**02M**