

NEWCHROM

SERIES 6700/6800

GAS

CHROMATOGRAPH

NEWCHROM TECHNOLOGIES

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INTRODUCTION

Series 6700 Gas Chromatograph is a Modular, Solid State Instrument designed for a variety of Analytical and Non-analytical applications.

It comprises of the basic unit to which a board range of optional modules may be added. Options available permit Isothermal or Temperature Programmed operation, use of Thermal conductivity or Flame Ionisation Detectors, syringe or valve sample introduction.

A large oven with a removable front opening door is standard. Each heated zone – Injectors, Detectors, Column Oven – is insulated to minimize heat transfer between zones, the Temperature of each one of which can be controlled and read independently.

Flow and Pressure controls of the Carrier Gas, Fuel and Air are housed on top towards the back of the oven. Top mounted Injectors allow vertical sample injection.

COLUMN OVEN: Here column is maintained at the required Temperature. It also houses the Injectors, Detectors, Flow Control Valves and Gas Lines. The Injector and Detectors have independent Heating Systems. Only the Detector in use is heated. The Oven interior contains the Heater Cage and the Temperature Sensing Element. The heater is a Newchrom Wire Element. The sensor is a Platinum Resistance Thermometer. The air inside the oven is intensely stirred by means of a blower for uniform temperature.

INJECTION PORTS

It consists of a stainless steel body which fits into a universal base and accepts a removable glass insert. For particular samples this glass insert may be packed with material which acts as a pre-column chiefly to protect expensive main column against dirt and high boiling components. The insert may be removed to be cleaned and packed again. The injector design ensures that the entire injector is swept with carrier gas, giving narrow band injection onto the column.

Liquid injection requires a microliter syringe. Gas injection requires either a gas tight syringe or a Gas Sampling Valve which introduces by pushpull mechanism, a fixed amount of sample from the sample stream into the carrier gas stream. The amount of sample introduced can be varied by changing the Sample Loop size.



DETECTORS

The FLAME IONIZATION DETECTOR yields excellent sensitivity and a wide linear dynamic range. The quartz jet permits the use of a total all glass separation system, and minimizes degradation and absorption of sample. Consequently, sensitivity and reproducibility in the analysis of drugs, hormones, steroids and other labile substances are increased.

The THERMAL CONDUCTIVITY DETECTOR is a non destructive universal detector. It is widely used in gas chromatography for its high reliability, simplicity and ease of operation.

The ICD measures the difference in thermal conductivity between the carrier gas flowing through a reference cell and a carrier gas / sample component mixture flowing through a measuring cell.

It uses the standard base which fits in the detector block of the gas chromatograph. Tungsten-Rhenium Gow MAC U.S.A. filaments have been built symmetrically into the detector housing.

Two filaments and four filaments configurations are offered. TCD Bridge current variable up to 300mA. Flow through type for high sensitivity. Internal volume less than 300 microliter.

ALL GLASS SYSTEM

Labile components which are readily absorbed, or react with hot metal surface require a completely internal all glass analysis path. In the Series 6700 GCs, the injection ports use glass inserts, the FID's have quartz jets. Therefore, together with glass columns, true all glass, highly inert separation systems are formed.



INSTRUMENT DESCRIPTION

SPECIFICATIONS:

COLUMN OVEN: Half Circular shape scientifically designed with double wall construction. Large volume. The oven can accept 1/4" or 1/8" metal or 1/4" glass packed, or capillary columns.

OVEN TEMPERATURE: Set by front panel mounted Thumbwheel Switches upto 399°C in 1°C steps.

INJECTOR DETECTOR TEMPERATURE: Set by front panel mounted Thumbwheel Switches up to 390°C in 10°C steps.

TEMPERATURE PROGRAMMER: Solid Stage Linear Temperature Programmer with rates 1,2,4,6,8,10,12,14,16,18,20°C/Min.

BUILT IN VOLTMETER: A.D.C. Voltmeter with range upto 20 Volts on a 3½" digit display is designed into the instrument for both internal and external use.

TEMPERATURE READOUT: Digital with 1°C resolution.

OVEN LIMIT CUT OUT: This is a safety system for oven that trips power to oven heater in the event of oven Triac or controller malfunction. This should be kept higher than normal maximum operation temperature.

TEMPERATURE CONTROL MODULE: This houses three independent control channels for the heating of:-

1. Oven Chamber
2. Injectors
3. Detectors – either T.C.D. or F.I.D.



The controller for the Oven, Injectors and Detectors is solid state and Proportional. The Neons starts flickering when equilibrium is attained. Sensing circuitry is isolated from the mains by means of a Trigger Pulse Isolated Transformer. The feedback of exiting temperature information is provided by Platinum Resistance Transformer that gives a large differential signal. Integrated circuit performs the logic function for control of Triacs placed in series with the Nichrome heater allow or the heater power.

A solid State Temperature Programmer is also provided. The programming rate can be selected say 1,2,4,6,8,10,12,14,16,18,20°C/Min. The selection switch has to be put in appropriate Isothermal or Programming mode, depending upon which one is desired.

THERMAL CONDUCTIVITY DETECTOR MODULE: This house the Electronics Thermal Conductivity Detector, A-4 core cable connects this module to the T. C. Detector. The Detector filaments are supplied power from and I. C. Regulated D.C. Power Supply about 18 V.

T.C.D. CONTROLS:

1. ON/OFF Switch.
2. Current Adjustable, Varies current from 80 mA to 300 mA.
3. Coarse and Fine Balance Controls: Facilities balancing of the system to pre-selected recorder baseline.
4. Attenuator in binary steps from XI to X1024 short position. The last position is marked Infinity and helps shorting of Recorder input terminals to select check Recorder Zero. The Linear binary attenuator helps produce on Recorder an expanded graphical representation of weaker signal and vice versa for stronger signal which are relative to the level of connection of component being analysed.
5. Polarity Switch. It reverses the polarity of the signal.



6. Mark Event: It is a ‘press to make contact’ type Switch upon press and release of which the Recorder pen would deflect 20-30 mm leaving visible mark on Recorder Chart. This facility is incorporated in the module to mark on Recorder Chart the event of injection of sample so that retention time of peaks could be directly calculated from the graph.

FLAME IONISATION DETECTOR MODULE: This house the Electronics for the essential requirements.

1. Power supply – Regulated to + 180 and – 180 Volt D.C.
2. Amplifier and its double ended I. C. Regulated Power supply \pm 12 Volts D.C. This is a high impedance high stability amplifier for measurement of very small currents.

F. I. D. CONTROLS:

1. ON/OFF Switch.
2. Sensitivity: In steps XI, X 10, X 100 and X 1000 the last position giving a selection 1000 times more sensitive than the first.
3. Attenuator in binary steps from X 1 to X 1024 and short position. The last position is marked infinity and helps shorting of Recorder input terminals to select / check Recorder Zero. The linear binary attenuator helps produce on Recorder an expanded graphical representation of weaker signal and vice versa for stronger signal which are a relative to the level of concentration of component being analysed.
4. Balance to be used for bringing base line to zero position.
5. Polarity Switch. It reverses the polarity of the signal.



6. Mark Event: It is a 'Press to make contact' type switch upon release of which the Recorder Pen would deflect 20-30 mm leaving visible mark on Recorder Chart. This facility is incorporated in the module to mark on Recorder Chart the event of injection of sample should that retention time of peaks could be directly calculated from the graph.

SAMPLE INTRODUCTION: Liquid samples are injected by means of a Microlitre Syringe and Gas samples by means of either a gas Tight Syringe or a gas Sampling Valve.

SPARES AND CONSUMABLES: Syringe, Septums, Valves, Special sample preparation and collection attachments for various applications, Columns, Fittings, Stainless Steel Tubing, Chart Paper, Ink etc. are available ex-stock with the company.

RECORDER : A potentiometer Strip Chart self balancing Recorder of Sensitivity 1 mV full scale deflection, Response Time around one second, chart width around 250 mm and High input Impedance is Recommended for Gas Chromatography work.

The following services are required for Installation of Gas Chromatograph:

1. Carrier Gas: The most widely used carrier gases are Nitrogen, Hydrogen, Argon and Helium. High Purity Gas cylinder with two stage Pressure regulator is essential.

2. Fuel and Air for F.I.D.: High Purity Hydrogen and Compressed Air cylinder with two stage Pressure regulators essential.

3. Mains Supply: 3 Nos. 2 pin 5 Amp and 1 No. 2 pin socket are required for Mains supply. There should be no leakage in Mains Supply and a good Earth point is essential. Power requirement is less than 1 KVA at 220 VAC. 10 50 Hz. In case the supply fluctuates or deviates more than 20 V (\pm 10 V), we recommended use of a Servo Type Stabilizer.

4. Soap solution capable of good bubble formation and a small point brush.

5. General Solvents. Small clean breakers or test tubes.



PRELIMINARIES: Carefully unpack all check against accompanying delivery note / challan. A convenient table height for placing / laying out the Instrument is 3 ft. For optimum performance the environment should be heat, clean free from air drought, high humidity and rapid /changes of ambient temperature. Wherever possible, or air conditioned room should be selected.

Mains supply wiring should conform to I.S.I. recommended configuration i.e. live on R. H. S., Neutral on L. H. S. and Earth on top of the socket looking from top.

The High Purity Gas cylinder should be tested for leakage and should be placed firmly adjacent to the instrument in a manner that makes it convenient to open and close the Gas supply. The Two stage Pressure regulators should be tested for leakage and proper regulation.

THEORETICAL DISCUSSION

Gas Chromatography is now an established technique for accurate, fast and convenient separation and detection of a mixture. The resolution of the Gas Chromatography Column and the level detection coupled with high speed response capability of the detectors make it the most widely used analytical technique. It is based on distribution of the sample component between two i.e. Mobile-Carrier Gas – Stationary – column packing. If the stationary phase is Liquid Chromatography and if it is solid, Gas Solid Chromatography.

QUALITATIVE ANALYSIS: Qualitative analysis (identification of the separated component) is based on the fact each component has a characteristics retention time for a particular set of operating conditions. The retention time of the component is measured along the base line from the injection point to the component is measured along the base line from the injection point to the apex of the component peak.

Tentative identification can be made by comparing previously recorded Chromatographs of know mixtures with by Chromatographs of the sample mixture. The know mixture is provided by including the components which are assumed will be present in the sample mixture.

QUANTITATIVE ANALYSIS: Quantitative analysis is based on the relationship between the peak parameters of height and the concentration of the component in the sample mixture. Quantitative analysis by peak height measurement alone can be accomplished when the sample mixture contains components with short retention times and when operating parameters can be duplicated precisely. However, most Quantitative analysis is accomplished by using peak area measurement as a basis.



To utilize peak area measurement efficiently for Quantitative analysis, a calibration curve necessary. The calibration curve is constructed by using known components as a standard.

Quantitative analysis is then made by the calibration curve in conjunction with peak area calculation.

PEAK AREA MEASUREMENT: Peak area measurement can be arrived at by a number of methods. Some of the common methods are (1) by multiplication of the height of the peak by the width of the peak at half height, (2) by multiplication of the height of the peak by the retention time, (3) by use of a planimeter can be used for peak area calculation, it is not practical for routine analysis since prohibitive amounts of time may be required.

HEIGHT – WIDTH: This is the most commonly used method. By this method, the area under the peak is calculated by multiplying the interpolated height of the peak by the width of the peak at half height (See Figure A).

The results are comparable to those obtained by integration when the peaks are Gaussian in shape. However, when unsymmetrical peaks are encountered, the calculated area does not correspond accurately to the actual peaks area: when very peaks are encountered, the peak width cannot be measured accurately enough for good results.

HEIGHT – RETENTION TIME: By this method, an area is calculated by multiplying the actual height of the peaks by the retention time (See Figure B). The resultant area (shaded area in Figure B) is proportional to the quantity of the component, therefore this area can be used similarly to the manner that the area peaks is used.

CARRIER GAS FLOW RATE: The flow rate of the carrier gas may be adjusted to provide optimum column efficiency. Column efficiency can be ascertained by observing the width of the peaks plotted on the strip chart or measured by the number of theoretical plates.

As a general rule, low flow and low temperatures will result in broad, flat peaks which yield unsatisfactory quantitative data. An increase in flow rate will tend to narrow the width of the peak, thus providing better resolution of adjacent peaks. The proper flow rate depends to some extent upon the column oven temperature, the volatility of the component in the sample mixture, and the column liquid phase.

As a good starting point, a flow rate of about 60 ml/min is recommended.



After a period of familiarization with the Gas Chromatograph, the most efficient flow rate can be more readily determined.

CALCULATION OF COLUMN EFFICIENCY: The optimum flow rate for a minimum theoretical plate height can be determined by plotting a curve of the height equivalent to a theoretical plate (HETP) versus the average flow rate of the carrier gas (u) as depicted in Figure C. Plotting points of higher and lower carrier gas rate (keeping all other parameters constant) will indicate the most effective flow rate for the column.

The height equivalent to a theoretical plate (HETP) is given by

$$\text{HETP} = L/N$$

Where L is the column length in centimeters and N is the number of theoretical plates. For a given column, a given sample size, and a fixed flow rate, the number of theoretical plates as calculated by

$$N = 16(x/y)^2$$

Where X is the distance from injection point to peaks apex y is the width of the peaks at the baseline.

SCHEMATIC AND DESCRIPTION

The basic parts of a Gas Chromatograph are shown in Figure D.

- a) Carrier Gas: To provide a stable gas flow through the column.
- b) Injector: For introduction and vaporization of sample.
- c) Column: Tubing packed with Stationary Phase.
- d) Detector: For measuring the quantity of separated components.
- e) Recorder: A display unit to provide permanent record of the analysis.
- f) Oven & Heaters: For temperature unit control of column, detector and injector.



Column length, inner diameter, and packing are important factors in achieving a required separation. Once this choice has been made, operating procedures (column temperature, flow rate, etc.) have to be adjusted to give the option compromise between component separation and analysis time.

For most analytical work, columns with inner diameters of 2 mm and O.D. 1/8" are readily employed, with lengths typically in the range of 3-9 feet (approx. 1-3 meters). Most general is a 2 Meter long column.

PACKING SELECTION: Literature generally available contains thousands of references to analytical separations, covering virtually every application

From this somewhat confusing range of possible column packing, it is possible to select one or more which should be suitable for at least the early stages of an investigation. Columns are generally supplied in packed form. An extensive reference library is maintained to provide assistance to users in the choice column for a particular application.

CONDITIONS: When a new or repacked column is installed in the column oven, it must be conditioned to equilibrate the liquid phases have a low vapour pressure; by conditioning, solvents and low molecular weight still present are bled out of the column.

Bleed-off contamination of the detector is reduced considerably by conditioning the column, that is, by operating the column for one to two days at slightly above the normal operating temperating temperature.

- a) Install the column in the oven, but double not connect the column outlet to the detector.
- b) Condition the column as described above.
- c) After conditioning, connect the column outlet to the detector.

Also make a leak test of the entire system.

SEPTUM REPLACEMENT: A worn septum will in the loss of sample due to vaporization "blow-black". After about 30 to 40 sample injections, the septum may require replacement. Replace the septum as follows:-



- a) Shut off the carrier gas temporarily using an external stop value; difficulties in accuracy reproducing flow settings are thus avoided.

NOTE: If the column is connected to a thermal conductivity detector; turn the POWER switch to OFF position to prevent possible oxidation of the DETECTOR filaments when the gas flow is interrupted.

- b) Remove the worn septum and replace with a new one.

NOTE:

1. Flame Ionisation Detector flames may extinguish following the procedure; check and relight if necessary.
2. When liquid phases sensitive to oxidation are used, always cool the column oven before opening the injection port.

THERMAL CONDUCTIVITY DETECTOR: The Thermal Conductivity Detector is based on the principle that a hot body will lose heat at a rate which depends upon the heat carrying capacity of the surrounding medium.

Thus, the rate of heat loss can be used as a measure of the type of gas. The detector contains two tungsten filaments which are heated, by a direct current. The filaments act as resistance elements for sensing changes in the thermal conductivity. A detector element and matching reference filament are opposed to obtain a differential signal. The heated elements are cooled by pure carrier gas and assure a definite resistance. As the mixture of eluted and carrier gas passes over the detector element, the mixture, because of different thermal conductivity, changes the rate of heat loss and the resistance of the element changes due to corresponding change in temperature.

The difference in resistance between detector elements is a function of the instantaneous concentration of the component in the gap stream. The reference and sensing resistance elements are incorporated into a Wheatstone bridge and the out of balance signal is applied to a recorder. It is a universal detector.

FLAME IONISATION DETECTOR: Flame Ionisation Detector operates on the principle that the Electronic Conductivity of gas is directly proportional to the concentration of charged particles within the gas. The sample component contained in the effluent gas from column is mixed with Hydrogen and burned in air. The extent of the ionisation depends on the nature of the compound its quantity and the temperature of the flame.

A collector electronic with a D.C. Potential applied is placed across the flame and measures the conductivity of the flame. With pure Hydrogen, the conductivity is quite low. However as compounds are combusted, the conductivity increases and the current which flows is amplified and fed to a Recorder. It is a Hydrogen Selective and very high sensitivity detector.

SELECTION OF OPERATING CONDITIONS FOR OPTIMUM OPERATION OF I.C.D.

Here we are presenting certain guidelines pertaining to determination of Optimum operating conditions; specific determination will depend upon nature of the sample.

CHOICE OF CARRIER GAS AND FLOW RATE: The following gases are recommended for use with this gas Chromatograph:

1. Hydrogen)
2. Helium)
3. Nitrogen) (Ultra High Purity; oxygen free)
4. Argon)

Choice of carrier gas will depend upon the component to be analysed. Hydrogen is the preferred carrier gas if it is not a component to be determined in the sample. Helium is the most recommended gas for many applications but is replaced by Hydrogen because of non-availability. Nitrogen and Argon have lower thermal conductivity and therefore lower current have to be employed and filament life reduces. However, if Hydrogen or lighter/ higher thermal conductivity elements are components to be detected, these gases are used.



DETECTOR SENSITIVITY, FILAMENT CURRENT AND FILAMENT AGEING: Detector sensitivity is dependent on choice gas, its flow rate,

filament current and oven temperature. Sensitivity is normally greater at low temperature and high bridge current. When using a new TCD, or one which has had new filaments fitted, the baseline of the recorder may drift in one direction during the first three days. This is an ageing effect of the filaments. The filaments will stabilise and the drift should stop within three or four days. After this period, the TCD may be used maximum sensitivity e.g. with attenuation at X 1 position, in conjunction with 1 mV Recorder.

SAMPLE SIZE: This largely depends on the analysis. It must be large enough to produce useful peaks for the component under study. The use of too large a sample can have a severe effect on column performance and give anomalous data. A sample size of 1 microlitre for liquids and 1 c.c. for gases is often a good starting point.

CAUTION: WHEN USING LARGE SAMPLE, ALWAYS REDUCE FILAMENT CURRENT TO AVOID FILAMENT DAMAGE.

SAMPLE INTRODUCTION : Clean the needle and syringe very well, insert the needle in the sample bottle and fill the syringe slowly, turn it upside down the plunger up slowly until the required amount of sample is left in the syringe. Wipe the needle clean and insert it immediately for its full length through the silicon rubber septum in the injection port. Inject the sample in one smooth quick press and withdraw the syringe.

NOTE: 1. The introduction of sample may cause the recorder pen to deflect, thus leaving a mark on the chart corresponding with the operation of injection.

2. Use needle of correct length. The needle must just reach the top of the column or just bottom of the injector.

3. Gaseous sample of suitable size may be introduction with either a gas tight syringe or by a gas sampling valve.



OVEN TEMPERATURE: The oven temperature is often fixed empirically. When partition column are used, a suitable oven temperature is usually within 50°C of the normal boiling point of the sample. In general, maximum resolution is obtained when the oven temperature is set at the normal boiling point of the component being analysed.

Although a reduced analysis time is gained by operating with a high oven temperature, too high an oven temperature can cause decomposition of the sample, excessive bleeding of stationary phase from the column, attack on the filament and reduced sensitivity.

INJECTOR AND DETECTOR TEMPERATURE: It has been found experimentally that injector temperature should be 25°C to 50°C above that of the oven for best results. Same for detector.

RECORDER SENSITIVITY: This model is designed for use with 1 mV potentiometric Recorder as standard.

OPERATING INSTRUCTION FOR USE OF THERMAL CONDUCTIVITY DETECTOR

When the Gas Chromatograph has been assembled on bench, connect suitable columns, between Injector and T.C.D. inlets oven, connect to the electrical and carrier gas supplies. Test for leaks in the gas lines and establish proper flows before switching the electrical power on.

LEAK CHECK: The presence of a leak is generally established by applying soap solution at the suspected point of leakage and in the event of a leakage the relevant nut is tightened by means of a spanner.

The sequence for pressure testing of a system for leakage is under:

1. Block the outlet of the exit tube. This can be done conveniently by using a cap union or merely blocking with a finger.
2. Raise the carrier gas pressure to about 10 1b over the working pressure.
3. After about 2 minutes, turn off the gas supply by rotating the pressure regulator knob fully anticlockwise or closing the shut off vane. The pressure gauge reading will decrease a little in the beginning but then should become steady.



4. Observe the pressure gauge. The pressure should decrease by not more than 1 psi in 15 minutes if there is no leak.

FLOW RATE ADJUSTMENT: To adjust the carrier gas flow through the column, connect the soap bubble flow meter to carrier gas outlet pipes with the help of connecting polyethylene the provide with the bubble flow meter. Open the gas cylinder and adjust the regulator to give about 2.5 kg/cm^2 pressure. Adjust the corresponding needle valve to give 30-40 ml/minute flow which is with a stop watch. The same procedure is followed to set same gas flow through the other column. To obtain good flow regulation, ensure that the pressure droop across the needle valve is at least 10 psi. Kindly note that one column will give a signal to one direction and the other in the opposite direction. Hence, the recorder baseline should be adjusted accordingly.

FILAMENT CURRENT CAUTION: Double not switch on the filament current until carrier gas has purged for at least 10 minutes; otherwise, the filament may be damaged. Make sure that the filament current adjust knob is set fully counter clockwise that the before switching on the bridge supply. It is important to remember that the filament should be operated at the lowest temperature (i.e. lowest the current consistent with the sensitivity required for the analysis).t lower current, the longer the filament life. Lower temperatures also reduce noise and increase stability, but note that sensitivity is approximately to the cube of filament current.

Note: Change in bridge current disturbs stability and therefore allow time until the base line stability is good before commencing analysis.

SWITCHING ON SEQUENCE: The following sequence MUST be observed for Gas Chromatograph.

1. Check that the thermal conductivity detector power supply is off, current to Minimum position and Attenuator to infinity.
2. Set the column flow rate of carrier gas supply and wait for about ten minutes.
3. Switch on the mains of Temperature Controller Module and the blower inside the oven should be on.
4. Set the oven temperature to bring the oven upto the proposed operating temperature.



5. Switch on the Injector and Detector heater and set the dial of the Injector and Detector controller for the required temperature.
6. The T.C.D. Module may now be provided the instrument has been purged for at least 10 minutes. Now set the bridge current to the valve.
7. Allow the system to stabilize. Bring the attenuator to the lowest setting which produces a straight baseline by means of Coarse and Fine Controls. The Chromatograph may be used for an analysis when the baseline is suitable. A trial analysis will indicate the correct setting for the output attenuator for achieving the desired Chromatograph, i.e. with all peaks on scale.
8. Balance the T.C.D. system and adjust.
9. Liquid sample can be injected through the injection port by piercing a septum on the injector port. A microlitre syringe is used for this purpose. Gaseous sample can be injected by gas tight syringe or a sampling valve. The septum is a self-sealing but after many injections the seal may become imperfect. It is important to use the correct replacement septum. Incorrect septum material will block the needle of the syringe; give bleed signal or other problems. Replacement septums are available Ex-stock.
10. Peaks which go off the scale may be brought on to scale by altering the Attenuator setting, and each peak should be marked with the setting on the attenuator switch used when it was traced. It is always desirable to record on Chromatography the instrument's settings. This permits subsequent comparison and repetition.

SWITCHING OFF SEQUENCE: Following sequence should be observed for switching off the instrument:

1. Switch off the recorder.
2. Reduce filament current to minimum and then switch off T.C.D. Module.
3. Switch off Injector and Detector Heating.
4. Lower the oven temperature setting and the oven door.



5. Allow the temperature of the oven and detector to come to almost ambient temperature.
6. Stop the carrier gas supply to the instrument only when column and detector temperature has fallen to near ambient temperature. For this shut off valve and finally close the cylinder with the help of cylinder key.

PRECAUTIONS:

1. Do not put On Mains without carrier. Switch on Mains only after 10 minutes of establishment flows.
2. Flow rate should be identical in both columns for best results.
3. Stabilized power supply should be used.
4. Instrument should be kept in a Dirt free clean place.
5. Use only High Purity Gases.
6. Use of Gas –Line Filters is preferable.
7. Do not Hard Push the syringe into injection port.
8. Keep our syringe clean by rinsing it with Pure Acetone or any other suitable Solvent.
9. Do not light a match – when using Hydrogen – to avoid any explosion because of some undetected leakage.

SELECTION OF OPERATING CONDITIONS FOR OPTIMUM OF FID

Here we are presenting certain guidelines pertaining to determination of Optimum operating conditions; specific determination will depend upon nature of the sample.

CHOICE OF GASES AND FLOW RATES: In F.I.D. generally, Nitrogen is used as a carrier, Hydrogen as fuel and air to support combustion. The flow rate of carrier has to be determined by the requirement of analysis. Hydrogen around 30 ml/min and Air around 300 ml/min for each flame.



IMPORTANCE OF CLEANLINESS: If the system is to be operated at high sensitivity, it is important to work at low a background current as possible; the column must, of course, have a minimum bleed rate, but, additionally the gases must be essentially free of Hydrocarbons and all parts of pluming must be clean. Tubing connections made with the plastic or rubber tubing should be avoided: It is a never-ending source of contamination. Polyethylene or nylon is only permissible type of tubing apart from clean stainless steel or copper tubing. It should also be remembered that once impure gases or dirty tubing has been used, all the internal plumbing (pressure gauges and needle valve, etc.) become contamination and a purge of as much as 2 to 4 days or more be required to clean up the system.

As an example of the importance and necessity of clean conditions, consider the case where some part of the system is contaminated with the equivalent of one drop of lubricating oil. Lubricating has a vapor pressure of one drop 0.01 mm the room temperature. This would contaminate the gases to a level of about 10 ppm. At a flow rate of 100 ml/minute, 10 ppm of lubricating oil would give background of about 10^{-11} amp. This is 10 to 100 times the background which can be tolerated for very high sensitivity. One drop of iol would require 167 hours to completely get out of the system at a flow rate of 100 ml/minute.

SAMPLE SIZE: This largely depends on the analysis. It must be large enough to produce a useful peak for the component under study. The use of too large a sample can have a severe effect on column performance and give anomalous data. A sample size of 1 microlitre for liquids and 1 c.c. for gases is often a good starting point.

SAMPLE INTRODUCTION: Clean the needle and syringe very well, insert the needless in the sample bottle and fill the syringe slowly, turn it upside down and move the plunger up. Slowly until the required amount of sample is left in the syringe. Wipe the needle clean and insert it immediately for its full length through the silicon rubber septum in the injection port. Inject the sample in one smooth quick press and withdraw the syringe.

NOTE:

1. The introduction of sample may cause the recorder pen to deflect, thus leaving a mark on the chart corresponding with the operation of injection.
2. Use needles of correct length. The needle must just reach the top column or just bottom the injector.
3. Gaseous sample of suitable size may be introduced with either a gas tight syringe or by a gas sampling valve.

OVEN TEMPERATURE: The oven temperature is often fixed empirically. When partition column are used, a suitable oven temperature is usually within 50°C of the normal boiling point of the sample. In general, maximum resolution is obtained when the oven temperature is set at the normal boiling point of the component being analysed.

Although a reduced analysis time is gained by operating with a high oven temperature, too high an oven temperature can cause decomposition of the sample, excessive bleeding of stationary phase from the columns and reduced stability.

INJECTION AND DETECTOR TEMPERATURE: It has been found experimentally that injector temperature should 25°C to 50°C above that of the oven for best results same for detector.

RECORDER: This is use for with 1 mV Potentiometric Strip Chart Recorder as standard.

OPERATING INSTRUCTIONS FOR USE OF FLAME IONISATION DETECTOR:

When the Gas Chromatograph has been assembled on bench, connect suitable columns between Injector and F.I.D. inlets inside oven; connect to the electrical, carrier gas, fuel and air supplies. Test for leaks in the gas lines and establish proper flow before switching the electrical power on.

LEAK CHECK: The presence of a leak is established by applying soap solution at the suspected point op leakage and in the event of a leakage the relevant nut is tightened by means of a spanner



FLOW RATE ADJUSTMENT: The flow of carrier and Hydrogen is measured by inserting/connecting Polythene tubing to the Quartz Jet while in cold condition. To adjust the carrier gas flow through the column, open the gas cylinder and adjust the regulator to give about 2.5 kg/cm^2 pressure. Adjust corresponding needle valve to give the required flow, say 40 ml/min taken on the soap bubble flow meter and timed with a stop watch. The same procedure is followed for the second column. Now open the carrier gas pressure regulator on carrier gas cylinder to stop the flow of carrier gas. The pressure will drop. For air supply a small compressor is supplied with the instrument which is connected via an I-joint to provide air for flame in both channels A and B. the air supply should be put off before next setting of Hydrogen. For very accurate work, we recommend use of Air Cylinder for air supply. If this is to be used restrictors are to be provided in series with each detector channel And & Be and then the air cylinder pressure regulator has to be adjusted to give 0.75 kg/cm^2 pressure. The set the flow rate of Hydrogen open the cylinder and adjust the regulator to read 2 kg/cm^2 . There is a needle valve in the instrument for Hydrogen. Adjust this to give an output pressure of 0.75 kg/cm^2 . Ahead of this are two restrictors which end in respective detectors. This will give desired flow to instrument; can also provide air for flames in both channels A & B. for precise work we recommend use of Air Cylinder only. If compressor is to be used, the air restrictors for both channel A & B are removed and replaced by blanks since the compressor cannot deliver air high pressure. To set the flow rate of Hydrogen open the cylinder and adjust the regulator to read 2 kg/cm^2 . There is a pressure regulator in the instrument for Hydrogen. Adjust this to give an output pressure of 0.75 kg/cm^2 . Ahead of this are two restrictors which end in respective detectors. This will give desired flow rate because the restrictors for channel And & Be are present. Now we can LIGHT THE FLAME. For this purpose use the lighter with the instrument. There will be a small sound as the flame is lit and the flame can also be observed by the naked eye. The same procedure is followed for Flame B. Please note the flame is lit with only Hydrogen on i.e. carrier and air off. Now we can open the shut off valve of Carrier and put on air. Close the cap of the detector. Please note that when the supply of air fails with the detector cover on, the flame will off pump is being used for air supply. When the work is over, to shut off flame first close Hydrogen supply, then air and lastly carrier. Please note carrier should not be stopped till the column is near room temperature.



SWITCHING ON SEQUENCE: The following sequences must be for switching on the Gas Chromatograph.

1. Check that all the switches are in off position.
2. Make sure that the column for rate of carrier gas supply, Hydrogen (fuel) for rate, and air for rate are correctly set.
3. Make sure there are no leaks.
4. Light Flame in both detectors and place detector caps in position.
5. Switch on the main of Temperature Controller Module and in position.
6. Set the oven temperature to bring the oven upto the proposed operating temperature.
7. Switch on the Injector and Detector heater and set the dial of the Injector and Detector controller for the required temperature.
8. Switch on F.I.D. Module.
9. Allow the system to stabilise. Bring the attenuator the lowest setting which produces a straight baseline by means of respective balance controls. The Chromatograph may be used for an analysis when the baseline is stable. A trial analysis will indicate the correct setting of the output attenuator for achieving the desired Chromatograph, i.e. with all peaks on scale.
10. Use in Isothermal or Programming Mode.
11. Liquid Sample carrier be injected through the injection port by piercing and septum on the injector port. A microliter syringe is used for this purpose. Gaseous sample can be injected be gas tight syringe or a gas sampling valve. The septum is a self-sealing but after many injections the seal may become imperfect. It is important to use the correct replacement septum. Incorrect septum material will block the needle of the syringe; give bleed signal or other problems. Replacement septums are available Ex-stock.
12. Peaks which go of the scale may be brought on to scale by altering the attenuator setting, and each peaks should be marked with the setting on the attenuator used when peaks was traced. It is always desirable to record on Chromatograph the instrument's settings. The permits subsequent comparison and repetition.



SWITCHING OFF SEQUENCE: Following sequence should be observed for switching off the instrument:

1. Switch off the Recorder.
2. Put Attenuator to Infinity position and switch off F.I.D. Module.
3. Close the Hydrogen shut off valve. The flame will go off. Shut off Sw air supply.
4. Switch off Injector and Detector Heater Power.
5. Lower the temperature setting of oven and open the oven door.
6. Allow the temperature setting of oven and Detector to come to almost ambient temperature.
7. Stop the carrier gas supply to the instrument only when the column and Detector temperature has fallen to near ambient temperature. For this close the shut off valve and finally close the cylinder with the help of cylinder key.

PRECAUTIONS:

1. Make sure there is no leakage in the gas system: special case is recommended for Hydrogen.
2. Flow rate should be identical in both columns for best results.
3. Stabilized mains power should be used.
4. Instrument should be kept in dirt free clean place.
5. Use only High Purity gases.
6. We recommended use of air cylinder for air supply specially for high sensitivity work, for low sensitivity work mini-compressor will suffice.
7. Use of Gas -line Filters is preferable.
8. Do not hard push the syringe into injection port.
9. Keep your syringe clean by rinsing it with pure Acetone.



MAINTENANCE-SERVICE & GENERAL PRECAUTION

ANALYZER OVEN: The front and rear motor bearing to be oiled after every few months. A good light machine oil may be used for lubrication.

If for any reason the motor is out of action, the temperature controller must not be switched on. Also critically check whether the fan blades are properly tightened and baffle approach to fan blade is firmly tight. If they are loose, they will create considerable noise and generate some vibration.

COLUMN:

- i) Columns should be handled with care to prevent abrasion of the support particles.
- ii) Columns should be stored in a dry place with the ends sealed by means of cap. They should be held vertically with the straight selection pointing upward in order to prevent tracking i.e. the creation of small passages along the sides of the packing material.
- iii) A sample even repeatedly injected will not usually contaminate the column or in any way affect its characteristics unless it contains non-volatile material.
- iv) Since some stationary phases tend to decompose thermally to some extent, it is preferable that the decomposition products should be continually removed. If the column is allow to remain at elevated temperature with no carrier gas flow, there will be and significant build-up of these products which may cause contamination of the Detector when the flow is restrictors-started. The condition may be corrected with continued passage of carrier gas but recovery time may be quite considerable. In the case of T.C. Detector, permanent damage may result. COLUMNS SHOULD NOT, THEREFORE, BE ALLOWED TO REMAIN IN A HEATED OVEN WITHOUT A FLOW OF CARRIER GAS. This effect can be aggravated if the columns stationary phase is oxidation sensitive, and therefore liable to suffer from oxidative breakdown as well as thermo decomposition, as oxygen may diffuse into the column in the absence of carrier gas flow.
- v) Due care must be taken to use carrier gases free from oxygen, moisture and hydrocarbons. The permissible level of oxygen in the carrier gases should be below 5 ppm and if it is more than that it will oxidise all polar columns and performance of these will keep on deteriorating from day-to-day and eventually column will be completely damaged and hence no resolution will be obtained. Our



polar columns are not guaranteed if Ultra High Purity gases of Indian Oxygen Ltd. are not used as carrier gas. It has been observed in our laboratory that an ordinary gas containing about 1% of oxygen is enough to oxidise and polar column within a period of 1 to 2 months. During initial testing full resolution was observed (5 peaks of a mixture of acetones, benzene, toluene and xylene at 90°C and finally single peak after the oxidation of the stationary phase).

FLOW MEASUREMENT ASSEMBLY: If the bubble flow meter and its connecting tubing is not cleaned and the soap solution (to be made only from liquid soap) is not charged periodically it will be extremely difficult to have a bubble formation. To enable measurement of the exact flow, it is recommended to clean this assembly every week and wetting of the glass surface of the flow meter before measuring the flow.

INJECTION

- i) The use of clean syringe is essential if anomalous results are to be avoided.
- ii) Cleaning of injection tube is also vital. The injection tube is often the highest temperature in the instrument and encounters the highest concentration of chemicals. It may, therefore, become contaminated with thermal degradation products. Charred residues, in particular are trouble-some and can give absorption effect which result in the ‘tailing’ of peaks.
- iii) After removing the injector top, it may be cleaned by flushing through with solvents. It may be found necessary to scrap off small particles.
- iv) After the cleaning operation, make sure that the side tube feeding the carrier gas is not blocked.



CLEANING OF TC DETECTOR: Decomposition products in the Detector block and on the filaments can upset the heat transfer properties of the filaments, change the zero balance and result in a noisy or drifting baseline. This trouble is not very common. However, it is possible for stationary phase from the column to enter the Detector block. Detector performance will then be affected and the exit tube may become blocked. There are several methods of cleaning the block. In the first method the Injector tube is also cleaned at the same time.

Replace the column by a short length of stainless steel tubing (protect the ends of the column whilst it is out of the instrument. Set carrier gas at 50 cc/min., raise oven temperature to about 150 °C. Inject 2 ml lots of distilled water through the septum. If the system is very contaminated, the droplets of water emerging from the exit will be dirty. Continue to make injections of water until the system is clean. Then raise the temperature to 250 °C and repeat.

FILAMENT: Filaments are very fragile and should, therefore be, handled with the greatest care. Avoid exposing them to dirt, dust or grease. Let these be handled by factory trained Engineers.

CLEANING OF F.I.D.: The normal maintenance problem with the Flame Ionization Detector is that carbon will be deposited on the collector as well on the jet. When the baseline noise becomes considerable, then it is suggested that the Detector should be cleaned. The F.I.D. is removed by disconnecting the column connection, electrical connection and jet assembly is unscrewed. Jet and collector are dipped in chromic acid for some time and after that they are thoroughly washed in water and cleaned with a fine zero energy paper. Finally they are washed with acetone and methanol and dried up in the oven. The Detector is then assembled as earlier, making sure that there is no Hydrogen leak while assembling it.



GAS CHROMATOGRAPHY SPARES SERVICE ACCESSORIES CONSUMABLES

INSTRUMENTS SERVICE / REPAIR APPLICATION

FOR USERS OF ANY MAKE INDIGENOUS OR IMPORTED GAS CHROMATOGRAPH And WIDE RANGE OF CHROMATOGRAPHY SUPPLIED ARE OFFERED BY NEWCHROM ENGINEERS.

COLUMNS

Prepacked Glass, Copper, Aluminum, Nickel, Teflon and Stainless Steel Columns with choice of Supports, Solid Absorbents and Stationary Phases.

SEPTA

Made out of high grade Silicon Rubber. Standard is 3/8" diameter 1/8" thick. Other sizes on request.

SYRINGES

Best International Hamilton Make Gas Tight Capacities 50, 100, 250, 500, 1000, 2500 Microliter.

Liquid Injection Capacities 1 Microliter, 10 Microliter, 50 Microliter.

DIGITAL PRESSURE MONITOR

Using a special Transducer, Pressure is converted to an Electronic signal which is processed and presented on a 3 1/2 digit display with unique features.

- Units selection: with a push button calibration in kg/cm² or p. s. i.
- Split second response time.
- High Accuracy of 1%.
- Pressure resolution of 0.1%.



Available in range 0-300 psi, 0-5000 psi etc. Useful in gas streams of Gas Chromatography, solvent stream of H.P.L.C. and general laboratory and Industrial measurements.

FITTING AND CONNECTORS

Swagelok Type made to international standards. Offered in Stainless Steel and Brass.

ADAPTOR

For connecting 1/4" columns to 1/8" part or connecting 1/8" columns to 1/4" part.

Union

For 1/16", 1/8", 1/4" tubing.

Reducing Unions

1/8" x 1/16"

1/4" x 1-1/8"

1/4" x 1/16".

Male Connector

1/8" T x 1/8" P

1/8" T x 1/8" P

1/4" T x 1/4" P

Nuts - 1/16", 1/8", 1/4"

Front Ferrules - 1/16", 1/8", 1/4"

Back Ferrules - 1/16", 1/8", 1/4"

Standard are Stainless Steel and Brass. Teflon and Graphite Ferrules also available.



RECORDER PAPER

We maintain stocks of recording paper for standard popular make Recorders. Please send paper sample with each enquiry.

GAS SAMPLING VALVE

For introducing Gas / Vapour Samples from Sample Stream into Carrier Gas System. Sample Size can be changed by changing sample loop.

Stainless Steel body with welded 1/8" fittings. Supplied with one 0.5 ml sample loop. Other loop sizes on request. Designed for panel mounting and general purpose.

FINE NEEDLE VALVE

For adjusting Flow of Nitrogen, Hydrogen, Air and other gases.

GAS PURIFIERS

Newchrom Gas Purifiers are designed to remove moisture, oil and other foreign materials from carrier gas stream. They are packed with Modular Sieve. There is a frit at the outlet to stop any fines from entering the flow system. We also offer purifiers packed with Activated Charcoal to remove organics that could cause problems.