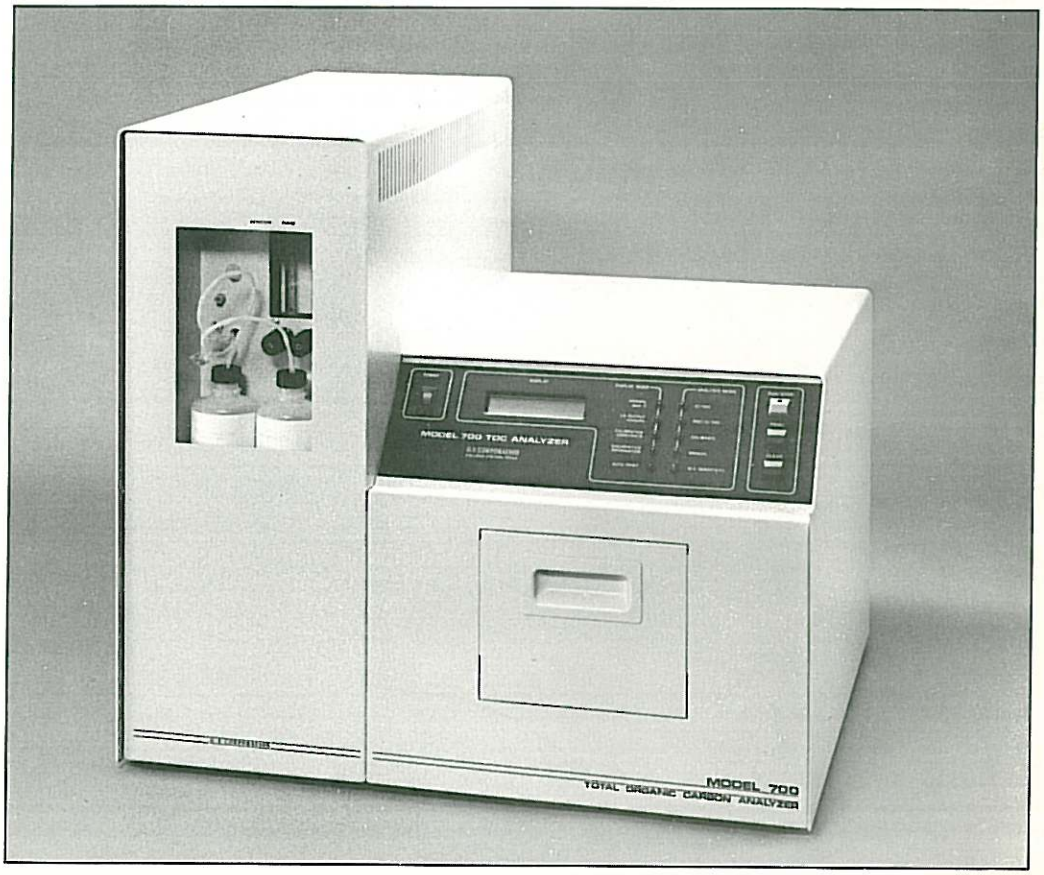
MODEL 700 TOC
TOTAL ORGANIC CARBON ANALYZER
USERS' MANUAL



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Chapter 1
Introduction

The Model 700 is a totally automated system for analyzing aqueous and solid samples for Total Organic Carbon, Total Inorganic Carbon, and Purgeable Organic Carbon. It takes advantage of O.I. Analytical's 16+ years of experience in development and use of the classic persulfate oxidation method for analysis of samples containing 4 ppb to 10,000 ppm of organic carbon. Its analytical range, accuracy, and precision are unmatched by any other analyzer presently on the market.

Definitions/Abbreviations

TC - Total Carbon: Defined as all carbon in a sample, including inorganic, organic, and volatile carbon, as they may be present. TC is reported in terms of total mass of carbon per unit of sample (mg C/L, etc.).

TIC - Total Inorganic Carbon: Defined as that carbon in a sample which is converted to carbon dioxide after acidification of the sample. TIC includes all dissolved carbon dioxide, bicarbonate, and carbonate species and is reported in terms of total mass of carbon per unit of sample (mg C/L, etc.).

TOC - Total Organic Carbon: Generally defined as that carbon in organic compounds which is converted to carbon dioxide by oxidation, after inorganic carbon has been removed or subtracted. Although TOC in water samples should ideally include carbon in volatile materials, most laboratories report TOC analyses of samples in which volatiles have been previously removed. In fact, the methods involving persulfate oxidation, which are widely accepted and used, each call for acidification and purging to remove inorganic carbon before oxidation of organics. This purging can also remove volatile organics before oxidation, though the results are still generally accepted as TOC (as they are with the Model 700 and in this manual). Volatiles can be included in TOC by separately measuring TC and IC and calculating TOC by difference.

DOC - Dissolved Organic Carbon: Defined as that organic carbon which is determined by analysis of aqueous samples which have been filtered through 0.45 micron filters. DOC is reported in terms of total mass of carbon per unit of sample (mg C/L, etc.).

SOC - Suspended Organic Carbon: Defined as that organic carbon which is determined by analysis of particles captured by filtration of aqueous samples through 0.45 micron filters. SOC is sometimes called Particulate Organic Carbon (POC) especially in the marine chemistry literature. It is reported in terms of total mass of carbon and referred to in this manual as SOC.

POC - Purgeable Organic Carbon: Defined as that organic carbon which is purged from solution by a stream of gas under a specific set of purging conditions. Specific conditions have not yet been standardized in the scientific literature.

POX - Purgeable Organic Halide: Defined as organic species containing any halogen (eg. Cl, Br, I) which can be purged from solution by a stream of gas under a specific set of purging conditions. Specific conditions have not yet been standardized in the scientific literature.

NPOC - Non-Purgeable Organic Carbon: Defined as that organic carbon which remains in solution after a sample has been purged by a stream of gas under a specific set of purging conditions. NPOC is often reported as TOC due to popular methods which require acidification and purging of TIC prior to oxidation of organics. This substitution is valid for samples containing negligible volatile or purgeable organic compounds (see also TOC defined in this chapter).

ppm C - parts-per-million Carbon: Defined as mass units of carbon per million sample mass units (ug C/g). In aqueous samples this is generally taken to be the same as mg C/L, as it is in this manual.

ppb C - parts-per-billion Carbon: Defined as mass units of carbon per billion sample mass units (ng/g). In aqueous samples this is generally taken to be the same as ug C/L, as it is in this manual.

Reagent Blank: Defined here as the detector response in millivolts generated from an analysis sequence (with reagents) without introduction of a sample or standard. The response is due to carbon contamination in the reagents, gas, reaction vessel, and/or tubing.

Standard: Defined as any sample to which a known amount of carbon has been added.

Standard Features
Syringe and Loop Injection

- The Model 700 TOC Analyzer can analyze aqueous samples for TOC in the range of 4 ppb C to 10,000 ppm C, and TIC and POC in the range of 1 ppb C to 10,000 ppm C, all on the same sample, with no sample pre-treatment, pre-purging, or dilution.

- The analytical range of the Model 700 depends on the volume of sample analyzed. Volume is selected by the analyst and is introduced by syringe injection or sample loop, and is dependent on the general range of concentration.

- The Model 700 can analyze samples that have high (near saturation) levels of dissolved solids including chloride. Thus, TOC in chemical solutions, reagents acids and caustics can be quantitatively determined.

- The Model 700 can analyze samples with suspended solids (up to 500 microns diameter) for TOC, so these samples need not be filtered prior to analysis. The method allows quantitative oxidation of carbon in the particulates, so a more accurate TOC may be reported.

- Spikes of known carbon mass may be added to samples in the instrument for "Method of Standard Additions" verification of TOC recovery in hard-to-handle samples such as acids, caustics, brines, and chemical reagents.

- Sample wetted parts consist entirely of fluorocarbon polymers (Kel F, TFE, FEP) to minimize carbon contamination during sample introduction, digestion and purging. They are also chemically compatible with essentially all solvents, acids, and bases.

- The single-beam photometric system in the infrared analyzer minimizes influence

due to contamination of measuring cell and vibration. It features dramatically improved long-term stability and signal-to-noise ratio over conventional dual-beam analyzers. The single-beam photometric system requires no delicate adjustment of optical balance. An optically-vertical dual-chamber detector effectively minimizes influence due to interfering or contaminant gas components.

- All electronic and mechanical components are exposed when the two instrument cowlings are removed for ease of maintenance and service. Red and green power status indicator lamps for each of the power supplies, valves, heaters, and other components driven by AC or DC voltages, are provided for ease of troubleshooting. All components have been positioned for quick access and replacement if necessary.

- The Model 700 is controlled by an 8085 microprocessor, which is used to regulate temperatures, control timing sequences, perform calculations, and perform continuous system diagnostics.

- Results of analyses are displayed on a screen directly as ppm C for TIC, TOC, TC, POC and ppm X for POX.

- The Model 700 features a linearized infrared analyzer so that a fast, single-point calibration may be used. This single-point calibration is used for TIC, TOC, TC, and POC determinations; therefore, separate standards are not needed.

- The Model 700 will retain in memory the conditions of analysis including blank values and the calibration constant for several years without external power. The user may select the ability to retain previous conditions of analysis or to return to default conditions upon power up.

- Conditions of analysis (times, temperatures, volumes, calibration constant, blanks, last analysis results) may be displayed on the screen at any time and may also be printed.

- The Model 700 can automatically sample from a bottle or a flowing stream by means of a sample loop. Thus, regular sampling of process streams and unattended replicate sampling from sample bottles can be performed. The built-in-timer may be set to sample and analyze at a specified time interval (such as once per hour) up to once every 24 hours.

- The Model 700 has several modes of analysis for a single sample, each of which can be selected by the push of a key. These include:

Analysis Mode	Time Of Analysis
TIC and TOC	8 Minutes
TIC Only	3 Minutes
TC Only	6 Minutes
TOC Only	7 Minutes

- Each of these modes of analysis may be used in combination with other features including the sampling loop, autosampler and printer.

- Options added to the Model 700 allow several more modes of operation which can be selected by the push of a key. These include:

Analysis Mode	Time Of Analysis
TIC, POC, and TOC	10 Minutes
POC Only	5 Minutes
Ampule Analysis	3 Minutes
Wafer TOC Analysis	8 Minutes
Purgeable Organic Halide (POX)	5 Minutes

- When applicable, each of these modes of analysis may be used in combination with other features including the sampling loop, autosampler and printer.

- A sample ID number which increments for each successive sample may be preset. A sample stop number may be preset in order to analyze a specific number of samples unattended before returning to the standby mode.

- Sample purge time and reaction time may each be extended for analysis of samples of a difficult nature.

- The Model 700 may be used to automatically sample and determine carbon dioxide and volatile organic carbon in gaseous or ambient air samples. By means of the sample loop and pump, gas or air samples can be analyzed at regular specified time intervals, and results reported in any appropriate units.

- The TIC determination can be automatically converted to appropriate units for carbonate alkalinity. Carbonate alkalinity can be determined on bottled samples or from a stream at regular intervals and printed in units of mg $CaCO_3$ per liter. In fact, TIC, TOC and POC can be reported in any desired units in a similar manner.

- The standard hardware of the instrument includes two alarm relays. One is triggered when the measured concentration of TIC or TOC exceeds a preset level. The other is triggered when the measured concentration of TIC or TOC decreases below a preset level. Concentration set points are entered from the keyboard, can be displayed on the screen, and are listed by the printer during the conditions-of-analysis printout.

Applicable Literature

Method Acceptance

The persulfate/100° C method for TOC is accepted by the EPA and by Standard Methods as a valid method for TOC. Pertinent references are:

Methods For Chemical Analysis of Water and Wastes, EPA-600/4-79-020, *Environmental Monitoring and Support Laboratory*, Method 415.1 (1979).

Standard Methods for the Examination of Water and Waste Water, 15th Edition, *American Public Health Assoc.*, Method 505, pp. 471-475 (1981).

Scientific Literature Base: The oxidation of organics by persulfate at 90-100° C remains the most accurate means of measuring Total Organic Carbon (TOC) described in the scientific literature. The best evidence that a given methodology is the choice of serious chemists is its literature base. We know of no other TOC method which boasts such an extensive reference library in refereed journals. Some pertinent references are listed here. Saline solutions and particulated samples, as well as

solutions with hard-to-oxidize materials, do not pose the recovery problem that exists with the low temperature-UV methods. Though no method can claim 100% recovery of all compounds in every sample matrix, the persulfate/100°C method exhibits the highest compound recovery efficiencies in the most common sample types (pure waters, waste waters, salt waters) of any presently existing method. Virtually all organic compounds dissolved in water are oxidized with efficiencies of 98% or better.

Barcelona, M.J. (1984) TOC Determinations in Ground Water, *Ground Water*, 22, 18-24.

Fredericks, A.D. and Sackett, W.M. (1970) Organic Carbon in the Gulf of Mexico, *Journal of Geophysical Research*, 75, 2199-2206.

House, D.A. (1962) Kinetics and Mechanisms of Oxidations by Peroxydisulfate, *Chemical Review,* 62, 185-203.

Leaderer, B.P. (1978) Summary of the New York Summer Aerosol Study (NYSAS), *Air Pollution Control Assoc.*, 28, Number 4, 321-327.

Menzel, D.W. (1967) Particulate Organic Carbon in the Deep Sea, *Deep-Sea Research*, 14, 229-238.

Menzel, D.W. and Vaccaro, R.F. (1964) The Measurement of Dissolved Organic and Particulate Carbon in Seawater, *Limnology and Oceanography*, 9, 138-142.

Williams, P.J. Leb (1969) The Wet Oxidation of Organic Matter in Seawater, *Limnology and Oceanography,* 14, 292-297.

Williams, P.M. (1969) The Determination of Dissolved Organic Carbon in Sea-water: A Comparison of Two Methods, *Limnology and Oceanography*, 14, 297-298.

Williams, P.M. (1967) Sea Surface Chemistry: Organic Carbon and Organic and Inorganic Nitrogen and Phosphorus in Surface Films and Subsurface Waters, *Deep-Sea Research*, 14, 791-800.

Wilson, R.F. (1961) Measurement of Organic Carbon in Seawater, *Limnology and Oceanography*, 6, 259-261.

Summary of Method

Total Inorganic Carbon (TIC): Is determined by the measurement of carbon dioxide released by acidification of a sample. As pH of the sample is lowered, carbonate and bicarbonate ions are converted to dissolved carbon dioxide. This carbon dioxide is purged from solution, concentrated by trapping, then desorbed and carried into a non-dispersive infrared analyzer (NDIR) which has been calibrated to directly display the mass of carbon dioxide detected. This mass is equivalent to the mass of TIC in the sample. Concentration of TIC is calculated by dividing this mass by the sample volume.

Total Organic Carbon (TOC): Is determined by the measurement of carbon dioxide released by chemical oxidation of the organic carbon in the sample. After the sample has been acidified and purged of TIC, sodium persulfate ($Na_2S_2O_8$), a strong oxidizer, is added. This oxidant quickly reacts with organic carbon in the sample at 100° C to form carbon dioxide. When the oxidation reaction is complete, the carbon dioxide is purged from the solution, concentrated by trapping, and detected as de-

scribed for TIC. The resulting carbon mass in the form of carbon dioxide is equivalent to the mass of organic carbon originally in the sample.

Total Carbon (TC): Is determined by the measurement of carbon dixoide released by the complete oxidation of all carbon present in the sample (inorganic and organic). For this analysis, acid and persulfate are added together with the sample at the beginning of the analysis and allowed to react for a specified time converting all carbon present to dissolved carbon dioxide. When the reaction is complete, the resultant carbon dioxide is purged from the solution, concentrated by trapping and detected as described for TIC. The resulting carbon mass detected in this analysis can be equivalent to one of two sums. Either the sum of the TIC + POC + NPOC or if the sample was preacidified and sparged before analysis, the carbon mass will be due only to the dissolved organic carbon (i.e. NPOC).

Purgeable Organic Carbon (POC): The POC measurement is accomplished by the addition of a purgeables trap and a furnace to the analysis train. As the sample is being purged of carbon dioxide from TIC, purgeable organic compounds are also removed from the water sample. These compounds are carried by the purge gas stream onto a purgeables trap packed with Tenax GC where they are trapped and concentrated (other or combination trapping substrates are available upon request). The carbon dioxide from TIC passes through the POC trap and is carried onto the molecular sieve trap. After a pre-determined purge time, the purgeables trap is placed in-line with a 800° C furnace and the IR detector. The trap is rapidly heated to 200° C to desorb POC. The desorbed organic compounds are carried into the furnace with added oxygen where they are oxidized on a cobalt, iron, nickel catalyst to carbon dioxide. The resulting carbon dioxide is detected and the corresponding POC concentration is displayed.

Purgeable Organic Halide (POX): POX measurement is accomplished by the addition of a purgeables trap and interfacing the Model 700 to the Model 4420 ELCD (electrolytic conductivity detector). Samples containing halogenated volatile organics are introduced into the instrument and purged with an inert gas to a trap packed with Tenax GC where they are trapped and concentrated. After a preset purge time, the trap is placed in-line with the pyrolysis reactor of the Model 4420 and heated rapidly to 200° C to desorb the volatiles off the trap. As the compounds pass through the reactor any halogens present are pyrolytically reduced at ca. 850° C in the presence of hydrogen reaction gas to the detectable species HX (where X is F, Cl, Br, I). The resultant HX is carried out of the reactor to a conductivity cell containing a deionized solvent where a change in conductivity due to HX is linearized by the 4420 electrometer. The Model 700 microprocessor monitors the ELCD output and provides a displayed concentration and printer output as ppm X (parts-per-million halide).

Ampule TOC (Solids): A liquid, sludge or powdered sample is placed in an ampule of 10 cc capacity, and a solution of acidified persulfate oxidant is added. The ampule is purged with oxygen to remove inorganic carbon and ambient carbon dioxide and then flame-sealed. Next, the ampule is heated to 90-100° C in a water bath or oven to oxidize the organic carbon. The carbon dioxide produced is then purged from the ampule onto a molecular sieve trap where it accumulates during purging. After a specified purge time, the trap is placed in-line with the non-dispersive infrared analyzer and is quickly heated to desorb the trapped carbon dioxide. The carbon dioxide is carried into the detector and is measured by comparison with standard gas injections. Analysis is automatic from the CO_2 purge step to calculation of carbon mass.

For solid samples containing organic species resistant to persulfate oxidation, an alternate, dry oxidation has been shown to be effective. After the sample has been weighed into an ampule, acid is added and purged to remove inorganic carbon. The sample is dried and a measured amount of pre-combusted cupric oxide powder is

mixed with the sample. The ampule is purged with oxygen to remove ambient CO_2, flame sealed, then placed in a furnace and heated to 550° C for 2 hours to promote oxidation. When the ampule is removed from the furnace, quantitation of CO_2 produced from organic carbon is performed as described for wet oxidation.

Non-Dispersive Infrared Analyzer: The infrared gas analyzer measures gas concentration based on the principle that each type of gas component shows a unique absorption spectrum in the infrared region. The IR analyzer contains an infrared light source, a beam chopper, a measuring cell and a detector filled with a gas mixture containing the gas component to be measured (CO_2).

The light source emits infrared light in all directions. The infrared light emitted backward is reflected and added to the infrared light emitted forward. The infrared light beam thus formed passes through the measuring cell and is partially absorbed or attenuated by any CO_2 present as a sample passing through. The beam then reaches the front chamber of the detector. Both the front and rear chambers of the detector are filled with a gas mixture containing the gas component to be measured (CO_2). The infrared light beam is partially absorbed in the front chamber and residual light is absorbed in the rear chamber, thereby increasing pressures in both the chambers. The front chamber pressure increases more than the rear chamber pressure because of a greater amount of radiation entering the front chamber (it attenuates radiation to the rear chamber), slight gas flow is produced through a path connecting these two chambers.

When the measuring cell contains an interfering gas component showing an infrared absorption spectrum overlapped with that of CO_2, the interfering gas component also causes pressure increases in the front and rear detector chambers. In such a case, however, the pressure increases are identical because the front chamber contains no interfering gas component to attenuate radiation. Thus, no gas flow between cells is produced when interfering gas is introduced to the cell.

Between the infrared light source and measuring cell is a chopper blade which rotates to interrupt the infrared light beam at regular frequency (10 Hz) so that it reaches the detector chamber intermittently. Therefore, pressure rises periodically in the chambers to produce a slight flow pulsation. The amplitude at the flow pulsation is greatest when no CO_2 is flowing through the measuring cell. The flow pulsation is converted into AC electrical signals by a micro-flow sensor located in the path connecting the chambers. The AC signals are amplified and rectified into DC voltage signals to be supplied to the microprocessor. The voltage output is linearized with respect to the mass of carbon (volume of CO_2) momentarily flowing through the cell by means of a third order polynominal equation. Specifications of the infrared analyzer are given later in this chapter.

Interferences

Method Interferences: Carbon is ubiquitous in nature, so reagents, water, and glassware cannot be cleaned completely of it. Method interferences (positive bias) may be caused by contaminants in the gas, dilution water, reagents, glassware, or other sample processing hardware such as homogenizers. All of these materials must be routinely demonstrated to be free from interference under the conditions of analysis by running reagent blanks. The use of high purity or purified reagents and gases help to minimize interference problems.

Interference in Calibration: Typically with most TOC instruments a correction for TOC in the dilution water used for calibration standards must be considered, especially for standards below 10 ppm C. The Model 700 has been designed to eliminate this problem. Various ways to calibrate the instrument are discussed in **Chapter 4.**

Interference By Non-CO_2 Gases: The infrared detector is sensitized to CO_2 and accomplishes virtually complete rejection of response from other gases which absorb energy in the infrared region. Trapping and desorption of CO_2 on the molecular sieve trap isolates this component of interest and allows the complete absence of interference in the system from gases other than CO_2.

Interference in Sampling: For most accurate analyses, sampling containers should be free of organic contaminants. Plastic bottles can bleed carbon into water samples, especially when they are new, or when they are used for low level samples (less than 200 ppb C). Any new bottles (especially plastic) should ideally be filled with clean water for a period of several days or boiled in water for a few hours before use. Pyrex bottles should be washed and muffled at 400°C before first use. Sample TIC and POC can be affected by exposure to the atmosphere. Sample TOC below about 50 ppb C can also be affected by atmospheric exposure. In these cases, sampling bottles should be kept closed when possible, and autosampler vials should be equipped with septa for needle-piercing by the autosampler.

Optional Equipment

Voltage Options

- **Voltage Converter:** OI Part #169-187. From 100 VAC to 115 VAC, 4 receptacle, 1 KVA maximum power output.

Data Handling Options

- **Strip Chart Recorder:** OI Part #172-495. Single pen, multi-speed drive, variable full-scale output. Specify line voltage and frequency. Includes 4 pens, 1 roll paper. Monitors analog output of IR, baseline, and peak shapes. This recorder is useful for diagnostics and as backup for hard copy of data.

- **Printer, Okidata Microline 182:** OI Part #178-897. For automatic printout of TIC, TOC, and POC values. Includes #168-923 interface cable and 1, #138-546, 400-sheet paper package. Prints out sample number, sample ID specified by user, time of day, millivolt response, micrograms carbon, and concentration in ppm C or ppb C for each sample, standard, and blank.

- **Data Handling Software (DHS-7):** OI Part #178-496. Includes 3 diskettes (programs and one back-up) and #179-655 RS-232 cable assembly for interfacing the Model 700 with the RS-232 option to an existing IBM PC (or compatible). The DHS-7 software collects and stores TIC/TOC/POC data by a memory resident program, thus allowing other programs to be used during data acquisition. Manipulation of the stored data provides for mean, standard deviation, coefficient of variance, graphic display as well as report generation.

- **DHS System I:** OI Part #178-988. Includes the DHS-7 software (see above) plus an IBM compatible computer and a printer cable for an Epson printer (or compatible). Operational requirements include the RS-232 option (see below) on the Model 700 and a printer.

- **Serial Communications Port, RS-232:** OI Part #169-070. The Serial Port Option provides the capability for remote communication with the instrument. The basic option includes the RS-232 serial port hardware and serial communications software that allows the total analysis results to be transferred to a remote terminal or host computer. Custom communications software priced on request. With the Serial Port Option and a telephone modem, the Model 700 makes it

possible to send TIC and TOC data from any remote monitoring location. In addition, complete remote control of the instrument is possible by connecting a CRT terminal to the serial port of the instrument, by either direct connection or through a telephone modem. All instrument keypad functions are available at the remote terminal, as are all display messages and analysis results. Messages and analysis results may be transmitted automatically on a timed basis, with each message change, at the end of each analysis, or only upon request from the remote terminal. The serial communications option also allows remote instrument testing and troubleshooting to be performed by the factory if a modem is connected to the instrument. Specifications of the serial communications port are given later in this chapter.

Extended Capabilities Options

Process Sampling Capability: OI Part #164-559. For retrofit addition to #161-620 basic Model 700 unit. Consists of a sample loop valve, valve actuator, peristaltic pump, associated tubing, and electronic controls built into the Model 700 to automatically actuate the components at preset times in the analysis cycle. With this option, a teflon sampling tube can be placed in a sample bottle and the Model 700 will automatically pump samples through a loop and inject the loop volume into the analysis system. In this manner, the instrument can continually analyze replicates out of the sample bottle until the sample line is placed in the next bottle. Sample loop volume can be changed (0.34 to 10 ml) according to concentration range of samples. The process option also allows the sample loop to be plumbed directly into a source of process water to be analyzed. Alarm relays can be triggered at preset high and low concentration limits. The analysis sequence can be set to run continually (readout every 8 minutes) or with a delay between analyses of up to 24 hours.

Microliter Sample Loop Capability: OI Part #172-784. For retrofit to #164-559 process sampling capability. Consist of a sample loop valve capable of sampling volumes from ca. 20 ul up to 10 ml. Sample loops included are nominal 50 ul, 100 ul, 300 ul and 500 ul volumes.

Multiplexed Process Sampling Capability: OI Part #169-088. User-specified number of sampling loop valves and custom software for multiplexing ultra pure water process streams into the Model 700 for rotating analysis. Includes #164-559 Process Sampling Capability plus additional valving. The Model 700 can analyze loop samples from each of any number of different process streams by multiplexing them into the analysis stream. This option is offered on a custom system basis in which the user specifies the sequence of multiplexed analyses.

Autosampling Capability: OI Part #169-012. 76-sample capacity, 14 ml sample size, with wash station, for unattended TIC/TOC/POC analysis. Includes #168-402 Autosampler Module, #164-559 Process Sampling Capability and #168-915 Interface Cable. Other sample capacities/sizes and #168-410 septum piercing options are available. Specifications of the autosampler are given later in this chapter.

Purgeable Organic Carbon (POC) Capability: OI Part #164-533. Purgeables trap, valving, and tubing built into Model 700 for same-sample analysis of POC, TIC and TOC. Allows the operator to screen samples for solvent spills and ground water contaminants and can be used as a possible screen for trihalomethanes, since the detection limit is better than 1 ppb C. Specifications of the purgeables capability are given later in this chapter.

Ampule (Solids) Capability: OI Part #164-541. Extends Model 700 capability for analysis of solids, sludges, ceramics, etc., by classic ampule method. Includes #132-894 Ampule Purging and Sealing Unit and #169-210 Ampule Breaking Accessory. Virtually any material, aqueous or solid, which can be placed in an ampule can be analyzed for TOC including all of the sample types listed below. Virtually any solid material which can be finely divided may be analyzed. Sample size may be varied such that TOC from 100 ppb C to 100% C can be determined. Suspended organic carbon (SOC) and dissolved organic carbon (DOC) may be separately determined by filtration. Specifications of the ampule capability are given later in this chapter.

Aqueous Samples: Pure water, waste waters, sludges, salt waters, oil contaminated waters, chemical reagents, acids, caustics and brines.

Solid Samples: Soils, sediments, crushed rock, fused silica, ores, well cores, particulates in suspension, particulates on filter paper, sand, ceramics, metals and catalyst materials.

Wafertoc Capability: OI Part #169-096. Extends Model 700 capability for analysis of semiconductor wafers. Includes #164-179 Digestion Chamber for 4-inch wafers, #164-161 Block Heater, and tubing connections. #164-187 Digestion Chamber for 6-inch wafers is also available.

Purgeable Organic Halide (POX) Capability: OI Part #174-227. Model 4420 Electrolytic Conductivity Detector, tubing and interface cables to the Model 700 microprocessor. Requirements are the POC option (see prior description) valve, trap and tubing. Allows operator to selectively detect volatile organic halides in waters for sample screening prior to gas chromatography analysis such as EPA methods 601 or 624 with a detection limit of 1 ppbX (or chloride equivalent). Specifications of the POX capability are given later in this chapter.

Kits

Hardware Kit, Model 700: OI Part #169-103. Kit of items not purchased separately. Consists of all screws, nuts, washers, and other hardware used on the Model 700.

Accessory Kit, Model 700: OI Part #168-816. For 1 year's operation of Model 700. Complete listing of components is given in **Chapter 7**.

Service Kit, Model 700: OI Part #169-111. Complete replacement kit of components. Including #169-103 Hardware Kit and #168-816 Accessory Kit. Complete listing of components is given in **Chapter 7**.

Reagents and Materials

Reagent Water: Distilled or deionized water containing TOC of less than 200 ppb C is recommended. Reagent water is available in 1 liter bottles from OI which is ultra-pure in TIC and TOC (OI Part #169-301).

Warning: Sodium Persulfate is a strong oxidizer and should be handled with extreme care. Always wear chemical eye, skin and clothes protection when handling.

Sodium Persulfate (100 g/L): OI Part #174-194 for 100 g of crystals. OI Part #178-848 for 500 g of crystals. Prepare a 100 g/L solution of sodium persulfate by dissolving 100 g $Na_2S_2O_8$ into reagent water (1 liter total volume). Stirring may be necessary but do not heat. Transfer a portion of this solution to the appropriate reagent bottle provided with the instrument. Place the lid on the reagent bottle but DO NOT tighten. Place the bottle in a microwave oven and heat until the solution just comes to a boil. Immediately tighten the lid and

immerse in water to cool. This procedure purifies the $Na_2S_2O_8$ solution by reducing (but not eliminating) TOC content of reagent water added during solution of the crystals. The cooled solution should then be purged with inert gas for several minutes to remove any CO_2 from oxidation of organics (the Model 700 provides reagent bottle purge lines). This pre-cleaned sodium persulfate solution is available in 1 liter bottles (OI Part #169-236). Shelf life is ca. 3 weeks.

Warning: Phosphoric Acid is a corrosive material and should be handled with extreme care. Always wear chemical eye, and clothes protection when handling.

Phosphoric Acid (5% vol/vol): OI Part #110-080 for 85% acid. Prepare a 5% by volume solution of phosphoric acid by adding 59 ml of ACS reagent grade 85% H_3PO_4 to reagent water (1 liter total volume). This solution is available in 1 liter bottles prepared in ultrapure water (OI Part #169-244).

The acid solution may be purified, if high organic contamination of the solution is suspected, by adding 10 cc of the persulfate solution and immerse vented container in boiling water for at least two hours. The persulfate will oxidize any TOC in the solution and then completely autodegrade in two hours at 100°C. The cooled solution should then be purged for several minutes to remove any CO_2 from oxidation of organics (the Model 700 provides reagent bottle purge lines). The decrease in reagent blank resulting from this procedure is not generally worth the purification effort unless the acid solution is found to be abnormally high in TOC.

CAUTION: Potassium Biphthalate is a chemical irritant and may cause eye burns. Avoid contact with eyes, skin or clothing by always wearing appropriate protection.

Potassium Biphthalate Stock Solution (1000 ppm C): OI Part #136-954 for crystals. Prepare a stock solution by adding 2.128 g of KHP (previously dried to constant mass at 110°C) into a 1000 ml volumetric flask. Dilute to volume with reagent water. Lower concentration standards can be prepared from this stock. It contains 1.0 ug C per ul. This standard solution is available in 10 ml crimp-top vials (OI Part #169-252). Shelf life is ca. 3 weeks. for TOC

CAUTION: Sodium Carbonate is a chemical irratant and may cause eye burns. Avoid contact with eyes, skin or clothing by always wearing appropriate protection.

Sodium Carbonate Stock Solution (1000 ppm C): OI Part #136-962 for crystals. Prepare a stock solution by adding 8.826 g of Na_2CO_3 (previously dried to constant mass at 110°C) to a 1000 ml volumetric flask. Dilute to volume with reagent water. Lower concentration standards can be prepared from this stock. It contains 1.0 ug C per ul. This standard solution is available in 10 ml crimp-top vials (OI Part #169-294). Shelf life is ca. 3 weeks. for TIC

POC Catalyst: OI Part #154-740. Consists of 10 grams of Co, Fe, Ni oxides on alumina catalyst pellets for high temperature oxidation of purgeable organic carbon fractions when that option is installed. Also needed for repacking of furnace tubes is the quartz wool packing (OI Part #144-501).

Syringes: For syringe injection of samples through syringe injection port, a syringe with a 2 inch needle is required.

- 25 ul, 2" OI Part #110-205
- 100 ul, 2" OI Part #110-221
- 2.5 ml, 2" OI Part #137-051
- 10.0 ml, 2" OI Part #169-335
- 50 ul, 2" OI Part #110-171
- 500 ul, 2" OI Part #137-069
- 5.0 ml, 2" OI Part #169-327

Gas Service:

Type - Nitrogen or helium, zero grade
Purity - 99.98%
Service pressure - 30 psig (207 kPa)
Consumption - 700 ml/min

Gas settings - Manual Mode (PV>, TV1>):
Purge - 6.0 on rotometer (80 ml/min)
Carrier - 5.0 on rotometer (375 ml/min)

Optional Equipment (Gases Required):
Ampule Option - oxygen, technical grade
POC Option - oxygen, technical grade
POX Option - hydrogen, ultra pure, helium carrier gas, ultra pure is required

Ascarite Gas Scrubber: OI Part #169-145 for scrubbing CO_2 from gas supplies of lower than 99.98% purity.

Gas Tubing: 1/8" OD x 0.063" ID teflon, copper or stainless steel to provide gas service to instrument. Galvanized tubing or plastic tubing other than teflon is not suitable due to CO_2 bleed.

Default Instrument Settings

System Configuration:

- Acid pump - enabled
- Oxidant pump - enabled
- Sample pump - disabled (for blanks)
- Sample loop valve - disabled (for blanks)
- Auto-run - enabled (for blanks)
- Auto-print - enabled
- Auto-sampler - disabled
- Auto start override - disabled
- TIC low alarm limit - 0
- TIC high alarm limit - 0
- TOC low alarm limit - 0
- TOC high alarm limit - 0
- Sample ID number - 01
- Sample stop number - 00
- Analyze TIC only - disabled
- Analyze TC only - disabled
- Ampule analysis - disabled
- Wafertoc analysis - disabled
- POC-only analysis - disabled
- POX analysis - disabled

Temperature Settings:

- Primary Trap - 200°C
- POC Trap - 180°C
- Digestion Vessel - 100°C
- POC Furnace - 800°C

Time Settings:

- Auto-repeat delay - 00:00:00
- Extended reaction - 00:00:00
- Extended purge - 00:00:00
- Time of day 00:00:00 (set as needed)
- Sample pump time - 7 seconds (sample pump time is automatically set to sample volume (ml) + 6 seconds)

Volume Settings:

- Acid Volume - 200 ul
- Oxidant Volume - 500 ul
- Sample Volume - 1.000 ml

Specifications

General Specifications-Model 700

Operating Mode: Separate determinations on same sample for Total Inorganic Carbon (TIC), Total Organic Carbon (TOC). Analysis is completely automatic after sample injection.

Method:

- TOC - Persulfate oxidation at 100° C, purge and trap carbon dioxide, NDIR detection.
- TIC - Acidification, purge and trap carbon dioxide, NDIR detection.

Range:

- TOC - 4 ppb C to 10,000 ppm C
- TIC - 1 ppb C to 10,000 ppm C

Precision:

- TOC - Greater of ± 2% or 2 ppb C
- TIC - Greater of ± 1% or 0.5 ppb C

Detection: Linearized non-dispersive infrared (NDIR) analyzer.

Readout: Digital display of TIC and TOC concentration in ppm C. Printout (optional) of sample ID, time of day, millivolt response, micrograms carbon, and ppb/ppm C.

Calibration: Single point by carbon dioxide injection or prepared standard introduction by sample loop or syringe injection.

Time of Analysis:

- TIC/TOC analysis, 8 minutes with default parameters.
- Spikes of known carbon mass may be added to samples in the instrument for "Method of Standard Additions" verification of TOC recovery in hard-to-handle samples such as acids, caustics, brines, and chemical reagents.

Sample Size: 0.34 ml to 10 ml

Utility Requirements:

- Power - 115 VAC, 50/60 Hz, 800 W
- Gas - Nitrogen or helium, 99.98% purity, (zero grade) at 207 kPa (30 psig)
- Consumption - 700 ml/min

Dimensions:

- 23.5 inches (60 cm) wide
- 22.5 inches (57 cm) high
- 21.5 inches (55 cm) deep

Weight: 123 lbs (56 kg) shipping weight

Voltage Options:

- 230 VAC to 115 VAC convertor
- 100 VAC to 115 VAC convertor

Data Handling Options:

- Strip Chart Recorder
- Printer
- Serial Communications Port
- Data Handling Software (DHS-7)
- DHS System I

Extended Capabilities Options:

- Process Sampling Capability
- Multiplexed Process Sampling Capability
- Autosampling Capability
- Purgeable Organic Carbon (POC) Capability
- Ampule (Solids) Capability
- Wafertoc Capability
- Purgeable Organic Halide (POX) Capability

OI Part Number:

- #161-620 Model 700 TOC Analyzer, Syringe Injection Only
- #169-343 Model 700 TOC Analyzer, Syringe and Loop Injection

Particulate Size: 500 microns

Specifications-Infrared Analyzer

Method: Non-dispersive single beam sensitized to CO_2

Range: 0 to 50 micrograms carbon as CO_2 (0 to 100 microliters CO_2)

Precision: ± 0.5% of full scale

Stability:

- Zero drift; ± 1% of full scale/24 hr
- Span drift; ± 1% of full scale/24 hr

Noise: Less than 0.5% of full scale

Ambient Temperature: 0 ° - 40 °C

Ambient Humidity: Less than 90% relative humidity

Response Time (90% of Final Reading): Electrical system: 2 sec at 1 liter per minute. Response of actual gas: within 15 sec.

Power Requirements: 115 VAC, ± 10% 60 Hz, 30 watts (provided internal to Model 700).

Output Signal: 0 - 1000 mV DC

Linearity: Better than ± 2% of full scale provided by best fit third order polynominal regression.

Materials of Gas-Contacting Parts:

- Measuring cell: Reflective Au Foil
- Window: CaF_2
- Piping: Polyethylene

Sample Gas Temperature: 0° to 55°C

Warm Up Time: Less than 15 minutes for stability satisfactory for analysis.

Specifications-Ampule (Solids) Capability

Method: TOC by persulfate oxidation at 95° C or dry oxidation with CuO catalysis at 550° C followed by purge and trap carbon dioxide, NDIR detection.

Range: 100 ppb C to 100% C

Precision: ± 5% from 100 ppb C to 1 ppm and ± 2% for 1 ppm C and above.

Readout: Digital display of TOC concentration in ppm C. Printout (optional) of sample ID, time of day, millivolt response, micrograms carbon, and ppm/ppb C.

Time of Analysis: Digestion of ampules requires 30 minutes per set for persulfate oxidation or 2 hours for dry oxidation. Time of a purging cycle for each measurement is 3 minutes.

Utility Requirements:

- Purging and sealing unit power - 115/230 VAC, 50/60 Hz, 400 W
- Gas - oxygen, technical grade

Dimensions: Purging and sealing unit - 16 inches (41 cm) wide x 43 17 inches (43 cm) high x 15 inches (38 cm) deep.

Weight: Purging and sealing unit - 50 lbs (23 kg) shipping weight

OI Part Numbers

- #164-541 Ampule Capability, includes the following:
- #132-894 Ampule Purging and Sealing Unit
- #169-210 Ampule Breaking Assembly

Specifications-Purgeable Organic Carbon Capability

Method: Purge and trap purgeable organics, oxidize at 800° C to carbon dioxide, NDIR detection

Range: 1 ppb C to 10,000 ppm C

Precision: Greater of ± 2% or 0.5 ppb C

Readout:

- Digital display of POC concentration in ppm C
- Printout (optional) of sample ID, time of day, millivolt response, micrograms carbon, and ppm/ppb C

Time of Analysis: TIC/POC/TOC mode takes 10 minutes per analysis (Adds 2 minutes to standard sequence per sample). POC-only takes 5 minutes per analysis.

Dimensions: Option is internal to base instrument

Gas Requirements: Oxygen-technical grade (99.98%) at 100 cc/min

OI Part Number: #164-533

Specifications - Purgeable Organic Halide Capability

Method: Purge and trap purgeable organics, pyrolytic reduction at ca. 850° C to hydrogen halide, electrolytic conductivity detection.

Range: 1 ppb X to 100 ppm X as HCl

Precision: Greater of ± 2% or 0.5 ppb X

Readout: Digital display of POX concentration in ppm X. Printout (optional) of sample ID, time of day, millivolt response, micrograms halide and ppm/ppb X.

Time of Analysis: Time of purging is 5 minutes per analysis.

Dimensions: Reactor furnace adds 4.5 inches (11.4 cm) to depth of base instrument. Stand alone ELCD module - 8 inches (20 cm) high x 8 inches (20 cm) wide x 10 inches (25.4 cm) deep.

Weight: ELCD module - 20 lbs (9.1 kg) shipping weight

OI Part Number: #174-227

Specifications-Autosampler

Nominal Line Voltage: 100 ± 10 VAC, 117 ± 15 VAC, 234 ± 30 VAC, 50/60 Hz

Line Voltage Noise Tolerance: ± 170% of nominal line voltage, 10 microsecond pulses at any phase angle

Power Consumption: 90 watts maximum

Ambient Temperature Range: 0° to 40° C

Humidity: Up to 100% relative humidity if connected to line voltage

Physical Size:

Cabinet:	Overall With Wash Station:
Depth - 8.9" (23 cm)	Depth - 10" (26 cm)
Width - 11.5" (29 cm)	Width - 11.5" (29 cm)
Height- 6.0" (16 cm)	Height - 17" (43 cm)

Weight: 22 pounds (10.4 kg)

Tube Capacity:

- 114 - 10 mm to 13 mm tubes
- 76 - 16 mm tubes, 17 or 18 mm vials
- 27 - 28 mm tubes (40 cc EPA vials)

Transit Times (Nominal line voltage)

Sample to Sample:

- Within Rack - Less than 0.5 seconds
- Rack to Rack - Less than 0.70 seconds

Sipper:

- Sample to Wash - Less than 0.5 seconds
- Wash to Sample - Less than 0.5 seconds
- Lower - Less than 2.5 seconds
- Raise - Less than 3.0 seconds

Manual Advance: Sample number 1 to sample 114 - Less than 35 seconds

Input-Output Connector: Single sub 'D' connector

OI Part Number:

- #169-012 Autosampling Capability, includes:
- #168-402 Autosampler Module
- #164-559 Process Sampling Capability
- #173-328-28 Septum Piercing Capability (option)

Specifications-Serial Communications Port

Electrical Interface: RS-232, 3 wire, signal ground, transmit, receive

Baud Rate: 110-9600 baud, switch selectable

Character Length: 8 bits

Stop Bits: 2

Parity: None

Maximum Transmission Rate to Instrument: 5 characters per second (with unit in remote control mode of operation)

Data Format: ASCII instrument control characters and analysis results

Connector: DB-25

Pinout: Pin 2 - receive
Pin 3 - transmit
Pin 7 - ground

OI Part Number: #169-070

Specifications-Printer, Okidata Microline 182

- Printing method - Impact dot matrix
- Printing speed - 120 characters per second
- Printing direction - Bidirectional/short line seeking
- Character size - Standard 9 x 9
- Line spacing - Default is 1/6-inch
- Column width - 80 columns
- Paper feed - Adjustable pin feed
- Paper thickness - 0.3 mm (0.012 inches) maximum
- Ribbon - Cartridge ribbon (black cloth)
- Ribbon life expectancy - 3 million characters
- MTBF - 4000 hours
- Print head life - 200 million characters
- Dimensions - 75 mmH x 355 mmW x 379 mmD
- Weight - 4.5 kilograms
- Power - 120 AC ± 10%
- Temperature-
  -Operating 5° C to 35° C (41° F to 95° F)
  -Storage-30° C to 70° C (- 22° F to 158° F)
- Humidity - Operating 10% to 80% (no condensation)
- Vibration-
  -Operating 0.25 G, 55 Hz (maximum)
  -Storage 0.50 G, 55 Hz (maximum)
- Interface - Centronics compatible, Serial RS232-C super speed (up to 19,200 baud), Serial RS422-A super speed (up to 19,200 baud)
- Synchronization - By externally supplied STROBE pulses
- Handshaking - By ACKNLG or BUSY signals
- Logic level - Input data and all interface control signals are compatible with the TTL level

OI Part Number: #169-004 for printer with cable, includes:

- #178-897 Okidata Microline printer
- #168-923 Printer interface cable
- #138-546 Printer paper, 400 sheets

Chapter 2
Description of Components

In **Chapter 1** some definitions and abbeviations used in this manual, as well as the basic concepts of Total Organic Carbon analysis are discussed. Also outlined are summaries of the methods used by the 700, and followed by its features and specifications.

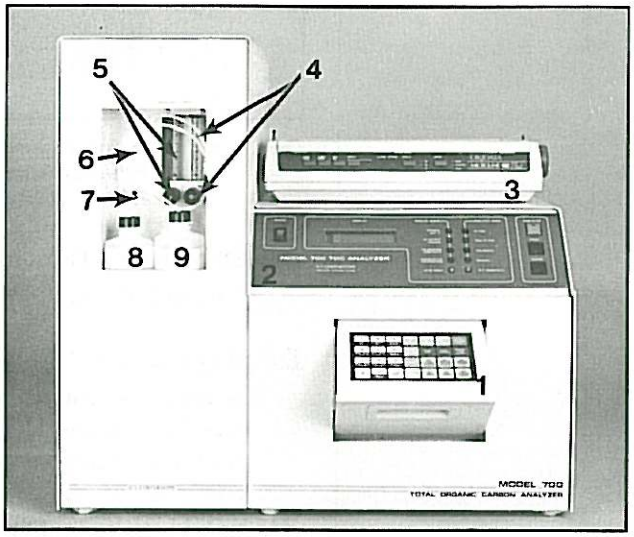
This chapter deals with what we have named the various components of the 700 and what the function of each is. Each significant component is pointed out and named on a photograph of one of the various views of the instrument. The function of each named component is also described, along with pertinant notes and cautions. The names are printed in **boldface type** in this chapter and are useful when you are trying to identify a part for ordering replacements.

Front View

Keypad: Used for entering and displaying the various types of information. Exposed by pulling outward on the fingerhold.

Display Panel: Displays various types of information.

Printer: Optional accessory (OI Part #178-897) automatically prints sample number, sample identification code, measured millivolts, measured micrograms and concentrations of samples and standards.



Front View
1. *Keypad*
2. *Display Panel*
3. *Printer*
4. *Purge Gas Flowmeter & Valve*
5. *Carrier Gas Flowmeter & Valve*
6. *Injection Port*
7. *Loop Sampling Inlet Port*
8. *Acid Bottle*
9. *Oxidant Bottle*

Purge Gas Flowmeter and Valve: Used for setting and monitoring proper flow ratio for the purge gas.

Carrier Gas Flowmeter and Valve: Used for setting and monitoring proper flow ratio for the IR detector gas.

Injection Port: Used for syringe injection of standards and samples. Threaded port fitting holds septum in place. Injection volume may range from 0.5 to 10 ml. After injection, acid rinses small-volume injections into the reaction chamber.

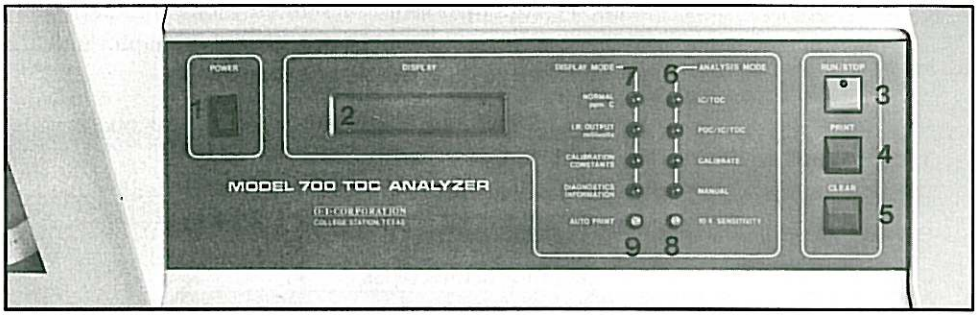
Loop Sampling Inlet Port: Inlet port for sample introduction by sample loop (OI Part#164-559). Sample may be aspirated into the sample loop through this

Luer-lok inlet port, or may be sampled intermittently from a flowing stream connected to this port.

Acid Bottle: Contains acid which is automatically pumped into the system during analysis. Bottle volume is 250 ml. Reagent line is 1/8" x 0.063" ID TFE fitted with a 10 micron stainless steel filter to remove particulates from acid solution.

Oxidant Bottle: Contains oxidant which is automatically pumped into the system during analysis. Bottle volume is 250 ml. Reagent line is 1/8" x 0.063" ID TFE fitted with a 10 micron stainless steel filter to remove particulates from oxidant solution.

Display Panel



Front Panel

1. *Power Switch*
2. *Display Screen*
3. *Run/Stop Key*
4. *Print Key*
5. *Clear Key*
6. *Analysis Mode Lamps*
7. *Display Mode Lamps*
8. *10X Sensitivity Lamp*
9. *Auto Print Lamp*

Power Switch: Turns main power to instrument and printer on and off. Should be illuminated when power is on.

Display Screen: Displays time elapsed from beginning of analysis, status of analysis sequence, instrument option messages, temperature setpoints, valve positions, and other diagnostic messages. Prompts analyst through calibration sequence with step by step instructions.

RUN/STOP Key: Starts and stops timer when pressed successively. Is pressed to begin analysis. Can be used to hold an analysis sequence at a particular time before continuing. Red LED is illuminated when system is running.

PRINT Key: When pressed, prints conditions of analysis (times, temperatures, volumes, etc.) and the results of the latest analysis. This key can be activated only when the timer is stopped.

CLEAR Key: Sets timer to zero when pressed. Used to reset instrument for the next sample analysis when auto-run is disabled or to interupt a current analysis sequence by placing instrument in the drain configuration and heating the trap(s).

ANALYSIS MODE Lamps: Indicates status of ANALYSIS MODE when illuminated.

DISPLAY MODE Lamps: Indicates status of screen display when illuminated.

10X SENSITIVITY Lamp: Indicates status of 10X SENSITIVITY option. This option is automatically activated 2 seconds before a CO_2 peak is desorbed, and

deactivated when NDIR millivolt response exceeds a raw output of 200 mV. Option is on when lamp is illuminated.

AUTO-PRINT Lamp: Indicates status of AUTO-PRINT option. Option is on when lamp is illuminated.

The capability to analyze samples for purgeable organic carbon or purgeable organic halide are optional features on the Model 700 (OI Part #164-533 and #174-227 respectively). If samples are analyzed using this mode without the POC option installed, TIC and TOC results will still be accurate, but no POC results will be displayed or printed. Since POX analysis requires the use of the Model 4420 ELCD, TIC, TOC and POC cannot be determined when the instrument is configured to run POX.

Keypad

SELECT ANALYSIS MODE Key: Advances ANALYSIS MODE through four options when successively pressed. Corresponding ANALYSIS MODE lamps on front panel will light to indicate present status of ANALYSIS MODE. Corresponding DISPLAY MODE lamps will light to indicate present status of the display screen. The message on the display screen will change accordingly. The four options for ANALYSIS MODE are described next.

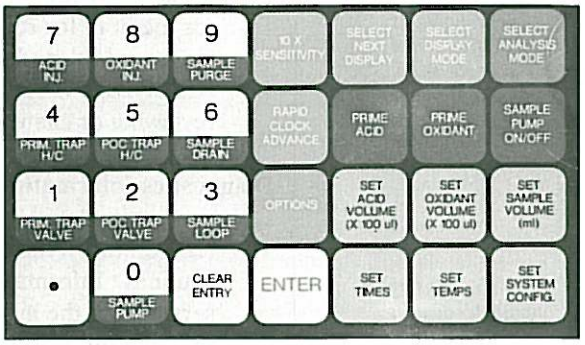


Fig. 2.3 Keypad

TIC/TOC Analysis Mode: Used for analysis of each sample for inorganic carbon (TIC) and organic carbon (TOC), and determination of corresponding reagent blanks. TIC-only or TC-only may be determined on each sample in this analysis mode when those particular functions are enabled (TOC-only may be determined in the Calibrate Mode).

POC/TIC/TOC Analysis Mode: Used for analysis of samples for purgeable organic carbon (POC) in addition to TIC and TOC. POC-only or POX analysis may also be analyzed.

Calibrate Mode: Used for calibration of the instrument. Calibration may be accomplished using a TIC standard or a TOC standard. The same calibration constant is used for TIC, TOC, and POC determinations (separate calibration of each is not necessary). The Calibrate Mode is different from the Analysis Modes only in that the screen display prompts the analyst through the calibration sequence. Thus, the Calibrate Mode may be used for sample analysis, and the Analysis Modes may be used for calibration. In this manner, samples may be analyzed for TOC-only using the TOC Calibrate option of the Calibrate Mode.

Manual Mode: Enables manual activation of valves, trap, heaters, and pumps. The components called out on the number keys at the left side of the keypad are activated by pressing the corresponding key when the instrument is in Manual Mode. The screen displays Diagnostics Information in this case.

SELECT DISPLAY MODE Key: Advances the DISPLAY MODE through the following options when successively pressed. The DISPLAY MODE affects only the information displayed on the screen. Any of the information described in this section may be reviewed (or changed when appropriate) without changing ANALYSIS MODE or affecting an analysis in progress.

Normal (ppm C): Displays on the screen the elapsed time and brief descriptions of successive events which occur during analysis. Displays carbon concentration

and corresponding linearized millivolt response at the end of the analysis sequence.

All instrument options are preset for routine analysis upon power up, and need not be reviewed or changed unless desired.

IR Output (millivolts): Display shows the continuous uncorrected (raw) millivolt output of the Infrared Analyzer. May be used when mechanically zeroing the IR and for monitoring baseline stability.

Calibration Constants: Displays on the screen, the values stored in the microprocessor memory for reagent blanks, mass (mg C) and response (mV) of the calibration standard, and scaling factor (ug C/mV) used for analysis as the SELECT NEXT DISPLAY key is successively pressed. This display mode can be used for reviewing or changing these values at any time including during an analysis.

Analysis using Calibrate Mode does not automatically change any of the calibration data.

Diagnostics Information: This display mode allows the analyst to view the system's current/updated valve positions, heated zone temperatures (as well as ambient temperature), the outputs of the A/D converters and temperature control routines. Information from this display mode is used for diagnostic analysis/servicing of the instrument and are available on seven sequential screens by pressing the Select Next Display key.

*The first screen displayed shows:

HT1<	TV1<	PV<	SV<
HT2<	TV2<	DV<	SP<

The code used here is:

HT1 = Heater 1 (heats primary trap)
HT2 = Heater 2 (heats POC trap)
TV1 = Trap Valve 1 (primary trap valve (A))
TV2 = Trap Valve 2 (POC trap valve (D))
PV = Purge Valve (turns sample purge gas ON/OFF)
DV = Drain Valve (sample drain valve (B))
SV = Sample Valve (sample loop valve (C))
SP = Sample Pump (to fill sample loop)
< = Deactivated (off)
> = Activated (on)

This screen displays the current status of these components. Components may be activated/deactivated with the instrument in Manual Mode by pressing the corresponding keys on the left side of the keypad.

*The second screen (press SELECT NEXT DISPLAY to advance screen) shows:

```
ADC1 DATA = XXXXX
ADC2 DATA = XXXXX
```

ADC1 DATA represents the DC voltage from the Infrared Analyzer. One count equals 0.5 mV in normal (1X) sensitivity mode. ADC2 DATA is the multiplexed analog/digital conversion of all temperature sensors (ambient temperature, primary trap, POC trap, POC furnace). Both displays are unscaled data ranging from 0 to 4095 counts.

*The third screen shows:

```
AMBIENT TEMPERATURE
00025   Deg. C
```

This is the temperature of the area immediately around the thermocouple MUX board. The ambient temperature is used for regulation and display of correct temperatures of heated components.

*The fourth screen shows:

```
TEMP HTR3 = 00025 C
TEMP HTR4 = 00025 C
```

Heater 1 is the primary trap heater. Heater 2 is the POC trap heater. This message displays the current temperature of these traps, regardless of the temperature set points.

*The fifth screen shows:

```
TEMP HTR1 = 00025 C
TEMP HTR2 = 00025 C
```

Heater 3 is the digestion vessel heater. Heater 4 is the POC furnace heater or ambient temperature if the POC option is not installed. This message displays the current temperature of these components, regardless of the temperature set points.

*The sixth screen shows:

```
TA1 = XXXXX    TD  = XXXXX
TI  = XXXXX    TDD = XXXXX
```

This display represents intermediate values of the Proportional/Integral/Differential (PID) temperature control algorithm. TA1 is the current actual temperature for the primary trap. TD is the instantaneous temperature error (proportional) between the set point and current temperature of the primary trap. TI is the integrated total of the temperature error over time. TDD is the slope (differential) of the error curve. The processor uses these values to regulate the trap temperature to the exact set point temperature and to rapidly heat the trap with negligible temperature overshoot.

*The seventh screen shows:

```
TA2 = XXXXX    TD  = XXXXX
TI  = XXXXX    TDD = XXXXX
```

This display represents intermediate values of the Proportional/Integral/Differential (PID) temperature central algorithm. TA2 is the current actual temperature for the POC trap. TD is the instantaneous temperature error (proportional) between the set point and current temperature of the POC trap. TI is the integrated total of the temperature error over time. TDD is the slope (differential) of the error curve. The processor uses these values to regulate the trap temperature to the exact set point temperature and to rapidly heat the trap with negligible temperature overshoot.

Successive pressing of the SELECT NEXT DISPLAY key causes the screen to scroll back through the same messages.

SELECT NEXT DISPLAY Key: Used to review information displayed on the screen when the DISPLAY MODE is in Calibration Constants or Diagnostics Information display modes. Successive pressing of this key causes an advance to the next information display on the screen and eventually to a repeat of the same sequence of messages. Press CLEAR ENTRY at any time to return to the beginning screen display.

CAUTION: Care should be taken not to add excessive amounts of reagent to the vessel when the instrument is not in the Drain configuration as liquid overflow from the digestion vessel can result with serious damage to the IR detector.

SAMPLE PUMP ON/OFF Key: Turns sample pump on and off at any time when pressed successively. After 1 minute, 10 seconds of an analysis, it may be used for manually rinsing the sample loop and tubing to minimize carry-over between samples with large differences in carbon content. Use of this key during the first 1 minute, 10 seconds of an analysis sequence is not recommended.

PRIME OXIDANT Key: Pumps 100 ul (1 pump stroke) of oxidant into the system each time the key is pressed. This key is always active when main power is on.

PRIME ACID Key: Pumps 100 ul (1 pump stroke) of acid into the system each time the key is pressed. This key is always active when main power is on.

The sample pump and sample loop are part of the Process Sampling Capability Option on the Model 700 (OI Part #164-559).

SET SAMPLE VOLUME (ml) Key: Used for telling the processor the volume of sample introduced for analysis. Volume is set by pressing this key, entering the correct sample volume in *ml*, and pressing ENTER. The sample volume may be set or changed at any time before or during analysis. The sample volume in memory may be reviewed at any time by pressing this key. Press ENTER to return the former screen display.

If solid samples are being analyzed by the ampule method, then entering the mass of the sample in grams will provide for carbon concentration in parts-per-million (ug C/g) to be calculated and displayed (or printed if the printer is being used) at the end of the analysis.

Actual volume injected is controlled manually by the analyst with syringe or by the sample loop (sample loops each have actual volumes labelled). The number of milliliters entered here is used simply for calculation of concentrations. If the volume set here in memory is different from the actual volume injected, the resulting display of concentration (ppm C) will be in error. In this case, the correct concentration may be manually calculated from the millivolt response or microgram C readout.

$$\frac{A \times B}{C} = D$$

where: A = Linearized IR Response (mV)
B = Scaling Factor (ug C/mV)
C = Sample Volume (mL)
D = Concentration (ppm C)

CAUTION: Care must be taken not to add a volume of reagent that will cause the total volume of acid, oxidant and sample to be greater than the digestion vessel (ca. 15 cc). Adding excessive amounts of liquid into the digestion vessel can cause system flooding and damage to the IR detector.

SET OXIDANT VOLUME (X 100 ul): Used for setting the volume of oxidant (100 g/L sodium persulfate) to be automatically dispensed during analysis. The volume is set at 1000 ul upon power up, which is sufficient for complete oxidation of most samples. The volume may be changed by pressing this key, entering the number of hundred-microliter pump strokes, then pressing ENTER. The metering pump delivers 100 ul per stroke. The maximum number of strokes which can be entered is 99 (9.9 ml).

Example: An entered value of 5 will cause the pump to stroke 5 times, to provide 500 ul of acid, during the appropriate time during analysis. The maximum number of strokes which can be entered is 99 (9.9 ml).

SET ACID VOLUME (X 100 ul Key): Used for setting the volume of acid (5% vol/vol phosphoric) to be automatically dispensed during analysis. The volume is set to 200 ul upon power up, an excess for sufficiently lowering the pH of most samples. The volume may be changed by pressing this key, entering the number of hundred-microliter pump strokes, then pressing ENTER. The metering pump delivers 100 ul per stroke.

Example: An entered value of 50 will cause the pump to stroke 50 times, to provide 5000 ul (5.0 ml) of oxidant, during the appropriate time during analysis.

SET SYSTEM CONFIGURATION Key: Used for setting the system configuration options. Each option may be reviewed by first pressing this key then repeatedly pressing ENTER. For user convenience, the System Configuration options are divided into three groups so that access to specific options in a group is made easier by pressing the key 1, 2 or 3 when prompted. Group 1 enables/disables components (pumps, valves, etc.) used for analyses. Group 2 consists of alarm set points and sample ID numbers. Group 3 consists of specialized analysis capabilities, such as TIC-only, ampule analysis, Watertoc analysis, POC-only, and POX-analysis. If no number is entered for a group when prompted, the listings on the screen begin with the first option in Group 1. Each option is

enabled when displayed on the screen by pressing 1 then ENTER, or disabled by pressing 0 then ENTER. Pressing CLEAR ENTRY will return the screen to the previous display mode.

Group 1

The terms Enabled/ Disabled rather than On/Off are used because enabling a particular option does not cause an immediate On/Off response such as when the sample pump key is pressed. Rather, options which are enabled turn on components automatically at the appropriate time in the analysis sequence.

Acid Pump: Enables/Disables the acid metering pump. The pump is generally enabled for sample analysis. Disabling is identical to setting the acid volume to zero and may be used for monitoring carrier gas contamination, if desired, by eliminating blank contribution from reagents.

Oxidant Pump : Enables/Disables the oxidant metering pump. The pump is generally enabled for sample analysis. Disabling is identical to setting the oxidant volume to zero and may be used for monitoring carrier gas contamination, if desired, by eliminating blank contribution from reagents.

Sample Pump : Enables/Disables the sample pump during the analysis. The pump should be enabled if sample is to be pumped through the sample loop for analysis.

The sample pump and sample loop are part of the Process Sampling Capability Option on the Model 700. (OI Part #164-559)

Sample Loop Valve: Enables/Disables the sample loop valve for sample introduction at the beginning of the analysis. Should be disabled for blanks and injection by syringe and should be enabled for sample loop injection.

Auto-Run: Enables/Disables the auto-run option. This function should generally be disabled for syringe injection and should be enabled for repetitive blanks and loop sampling. Auto-run can be disabled to end a sequence of replicates. When auto-run is enabled, a time delay between analyses may be entered.

The Autosampling Capability is an option on the Model 700 (OI Part #169-012).

Autoprint : Enables/disables the printer. When enabled, data are printed for each sample analyzed. It is important to note that when this function is enabled and a printer is connected to the Model 700, the processor receives diagnostic information from the printer. Thus, if a printer malfunction occurs (printer unselected, runs out of paper, etc.) during an analysis, the processor will automatically stop the Model 700 at the end of that run to prevent subsequent data from being lost. After the printer malfunction has been corrected, pressing the CLEAR button will allow the Model 700 to continue analysis sequences.

Autosampler : Enables/Disables TTL output commands to the autosampler. These commands raise and lower the autosampler sipper tube, move the sipper tube between the sample vials and the wash station, and advance the sample tray.

Caution: TOC analyses performed with the override option enabled should be considered accurate only if the user verifies that the actual digestion vessel temperature is 100°C.

Ready/Standby Status Override : Allows override of the Model 700 temperature check program. When the instrument is turned on, about 10 minutes are needed for the digestion vessel heater block to heat to the proper temperature for TOC analysis in the normal mode of operation. During this time, the RUN/STOP key may be pressed, and the analysis sequence will automatically begin when the block has reached temperature. The READY/STANDBY OVERRIDE option allows the user to override the instrument check program which disallows analysis without proper digestion block temperature. This option is useful in Ampule Analysis or Wafertoc mode when the digestion vessel heater is turned off or if servicing the instrument when the system standby prevents operation.

Group 2

Sample ID Number: Allows the entry of an ID number to correspond with printout of sample results. This number increases by one for each subsequent analysis.

Sample Stop Number: Stops repetitive analyses when the sample ID number reaches the number set here. This option allows the user to set a specific number of analyses to be performed during auto-run analysis or when using the autosampler.

Number of Reps Per Sample: Used primarily with autosampler option to allow for repetitive sampling from a sample vial. If the number of reps per sample is set to a number greater than 1, the sample ID number will remain constant (and be printed) for the number of reps set.

TIC Alarm High Limit: Energizes the High Alarm Output Relay #1 when the measured TIC concentration becomes equal to or greater than the value displayed here. The normally open (N.O.) terminal is shorted to common (COM.) when energized.

TIC Alarm Low Limit: Energizes the Low Alarm Output Relay #2 when the TIC concentratin decreases below the value displayed here. The normally open (N.O.) terminal is shorted to common (COM.) when energized.

TOC Alarm High Limit: Energizes the High Alarm Output Relay #1 when the measured TOC concentration becomes equal to or greater than the value displayed here. The normally open (N.O.) terminal is shorted to common (COM.) when energized.

TOC Alarm Low Limit: Energizes the Low Alarm Output Relay #2 when the TOC concentration decreases below the value displayed here. The normally open (N.O.) terminal is shorted to common (COM.) when energized.

Group 3

TIC Only: Enables/Disables the option to determine TIC only. Can only be used in TIC/TOC Analysis Mode. When enabled, the analysis sequence is modified to only determine inorganic carbon by acidification only.

TC Only: Enables/Disables the option to determine TC only. Can only be used in TIC/TOC Analysis Mode. When enabled, the analysis sequence is modified to determine total carbon by addition of acid and oxidant at the same time. This option can be used to determine TOC-only if samples contain no appreciable TIC or if they have been pre-acidified and purged of TIC.

Ampule Analysis: Enables/Disables the option to determine TOC by the ampule method. Can only be used in TIC/TOC Analysis Mode. When enabled, the analysis sequence is modified and shortened to automatically purge, trap, desorb, and detect CO_2 from ampule digestion of TOC. The Model 700 also calculates and prints the number of micrograms of carbon purged from the ampule.

Wafertoc Analysis: Enables/Disables the option to determine TOC on the surfaces of semiconductor wafers or any other substrate which will fit in the Wafertoc digestion chamber. Can only be used in TIC/TOC Analysis Mode. When enabled, the analysis sequence is modified to add acid and oxidant to the Wafertoc chamber, digest the wafer surface TOC, purge the resulting CO_2 , and calculate the number of micrograms carbon detected.

POC-only can be enabled simultaneously with any one of the other Group 3 options. In this case, the SELECT ANALYSIS MODE key determines which of the two enabled analysis options is used.

These are temperature set points. Actual temperatures may be monitored by using the Diagnostics Information DISPLAY MODE.

POC-Only Analysis: Enables/Disables the option to determine POC only. Can only be used in the POC/TIC/TOC Analysis Mode. When enabled, the analysis sequence is modified to determine POC by purging, concentration or trapping on a POC trap (Tenax GC), thermal desorption, combustion to CO_2, and detection.

POX-Analysis: Enables/Disables the option to perform POX analysis. It can be operated only when in POC/TIC/TOC analysis mode and the system is modified with the Model 4420 ELCD. When enabled, the analysis sequence will allow selective determination of POX by purging and concentrating (trapping) the volatile fraction of the sample on a trap packed with Tenax GC, then thermal desorption to the Model 4420 reactor and detector.

Since the output signal of the Model 4420 is linerized internal to the detector when the POX analysis is enabled, the Model 700 processor does not interpolate the signal as it does with the IR output signal when running TIC, TOC or POC analysis. Thus, the analyst should be certain of the system configuration when switching from POX analysis to organic carbon analysis and vice versa.

SET TEMPERATURES Key: Used for changing the temperatures of the digestion vessel, trap desorption, and the optional POC trap and furnace. Default temperature set points may be reviewed by pressing this key then ENTER, and may be changed (within limits) by pressing this key, entering the desired temperature, then pressing ENTER. Press CLEAR ENTRY at any time to return to the former screen display.

SET TIMES Key: Used for setting the following options regarding time. Preset time options may be reviewed by pressing this key then ENTER, and may be changed by pressing this key, the desired 6-digit time (hh, mm, ss) then ENTER. Press CLEAR ENTRY at any time to return to the former screen display.

Auto-Repeat Delay: Used for setting a time delay between analysis starts. The time entered in this function will be the time elapsed from the start of one analysis run to the start of the subsequent analysis run. An auto-repeat time of zero (default value) will cause the instrument to start the first analysis immediately following the end of second analysis and so on. The minimum auto-repeat time that can be entered is the time required to run one complete analysis plus one second (example: TIC/TOC is normally 8 minutes, 5 seconds, so auto-repeat must be at least 8 minutes, 6 seconds). This function is useful for on-line sampling of continuous process streams. It offers no advantage for syringe-injected sample or auto-sampler analyses.

Example: A delay time of 00:30:00 calls for analyses to be initiated every 30 minutes.

The primary trap is automatically activated (heated) 2 minutes prior to the next analysis start in order to bake out any contaminants accumulated during the delay period. If the IR output is being monitored by a chart recorder, a small peak resulting from this bakeout may be present on the chart record before each analysis period. This peak does not affect the analytical results.

Extended Digestion Time: Used for extending the time of reaction for oxidation of TOC.

Extended Purge Time: Used for extending the time of purging for TIC removal and trapping.

The sample pump and sample loop are part of the process Sampling Capability Option on the Model 700 (OI Part #164-559).

Time Of Day: When properly set, allows the printing of the correct time of day for each analysis. The 24 hour clock format is used for setting and printout.

Example: One-thirty pm is 13:30:00.

Sample Pump Time: Used for setting the time that the sample pump runs while pumping a sample through the sample loop. The pump rate is 1 ml/sec, so the volume pumped in milliliters is equal to the number of seconds set. The pump time will automatically default to the integer value of the sample volume setting (set by the analyst) plus 6 sec to allow a flush volume of at least 6 ml. This default setting can be changed at the analysts discretion to save sample (decrease time), or to assure proper loop rinsing from sample to sample (increase time).

10X SENSITIVITY Key: Turns On/Off the 10X amplification of the IR output. Allows for another digit of resolution in the millivolt output of the IR. May be used when the detector response to samples is less than 200 mV (20% of full scale). This option will automatically come on 2 seconds prior to peak detection and cut off when a 200 mV response is exceeded.

RAPID CLOCK ADVANCE Key: Advances timer at a rate 5 times faster than normal. Is turned on/off with successive pressing.

OPTIONS Key: Not active. Available for future software enhancements.

ENTER Key: Used for entry of numerical data. After any number is keyed in, the ENTER key must be pressed to enter the number into memory. This key is also used to advance the screen display if a currently displayed number is to remain in memory.

CLEAR ENTRY Key: Used to clear a number which has been keyed in but not ENTERed. Also used to return the screen to a former display mode when reviewing data in memory.

DECIMAL POINT Key: Used as normal decimal point for numerical data entry. Also used to display last analysis data when this key is pressed at times other than during data entry (data entry is prompted by any of the six SET keys or by display of calibration constants), the last analysis results in ppm C are displayed for 5 seconds. If pressed again before the former display returns, the last analysis results in millivolts are displayed. In this manner, results may be retrieved even after a new analysis has begun.

When determining multiple parameters per sample (TIC/TOC or POC/TIC/TOC) analysis results are updated for each parameter immediately after each peak is measured. Thus, the last analysis results displayed by pressing the decimal point key during a run may actually represent the TIC and/or POC value from a new sample and the TOC value from the previous sample, because TIC and POC are determined several minutes before TOC on a sample.

Keys 0-9 can be used to activate and deactivate the 700's mechnical components when in the Manual Mode. This is often useful for troubleshooting.

Number Keys 0 Through 9: Used for numerical data entry. In Manual Mode these keys activate/deactivate the components corresponding to the component names written on the bottom of the number keys.

Left Bay

Sample Pump: Used to aspirate sample through the loop sampling inlet and the sample loop. The pump should be by-passed during pressure-fed flow-through (on-line) sampling to avoid creating back pressure. By-passing the pump is accomplished by connecting the inlet tube of the pump (valve C, port 5), directly to the sample waste line (outlet of pump) using a spare 1/4"-28 coupling from the pump "legs" to operate the threaded fittings. This pump is included in the Process Sampling Option (OI Part #164-559).

Digestion Vessel Heater Block: Used for heating the digestion vessel by means of heater cartridges positioned in the block. Power to the block is controlled by the processor and is indicated by a red LED on the I/O board. Temperature of the block may be changed by using the keypad and may be displayed on the screen.

Digestion Vessel: Kel-F vessel in which samples are acidified, purged, and digested with oxidant while being heated.

Condensation Chamber: Used as a gross water condensor during sample purge.

Injection Port Block: On this fitting is mounted the injection port and septum, the acid feed line, and sample lines to the digestion chamber. Acid is fed into this fitting in order to rinse small volume injections from the injection port into the digestion vessel.

Purge Valve: A 2-way (on/off) valve for the purge gas. It is controlled by the processor during analysis and may also be manually controlled. It is rated at 100 psi.

Activated Carbon Scrubber: Used to trap and filter any organic contaminants from the purge gas line so they will not be oxidized in the digestion vessel and trapped during sample purge.

Purge Gas Check Valve: In line, one way valve to prevent back flow of contaminants in purge gas flow train.

Oxidant Pump: Pumps 100 ul of oxidant each time it is energized (12 VDC). It is controlled by the processor during analysis and may also be manually controlled. The inlet of this pump has a one way liqud flow valve to prevent back flow of reagent.

Acid Pump: Pumps 100 ul of acid each time it is energized (12 VDC). It is controlled by the processor during analysis and may also be manually controlled.

Furnace: Filled with an oxidation catalyst and is used for oxidation of purgeable organics to CO_2 after they are desorbed from the purgeables trap. This furnace is part of the Purgeables Option (OI Part #164-533).

Primary Trap (Innermost): A molecular sieve column for trapping carbon dioxide.

Purgeables Trap (Outermost): Used for trapping volatile organics purged from sample solution. Heating and cooling of the trap are controlled by the processor and may also be manually controlled. This Tenax trap is part of the Purgeables Option (OI Part #164-533).

Primary Trap Valve (Innermost): Rotates the primary trap between the purging/trapping and desorption/detecting positions. It is controlled by the processor and may also be manually controlled. This valve is also referred to as valve A.

Purgeables Trap Valve (Outermost): Rotates the purgeables trap between the purging/trapping and desorption/detecting positions. It is controlled by the processor and may also be manually controlled. This valve is part of the Purgeables Option (OI Part #164-533). This valve is also referred to as valve D.

Drain Valve (Innermost): Selects purge mode or drain mode of the digestion vessel. It is controlled by the processor and may also be manually controlled. This valve is also referred to as valve B.

Sample Loop Valve (Outermost): Rotates the sample loop between the fill-loop and loop-inject positions. It is controlled by the processor and may also be manually controlled. This valve is part of the Process Sampling Option (OI Part #164-559). This valve is also referred to as valve C.

Actuator Gas Inlet: Provides gas (air or nitrogen) for pneumatic actuation of the valves. Thirty psi gas pressure is required for proper valve rotation.

Nitrogen Gas Inlet: Provides nitrogen gas flow for carrier and purge gas, IR case purge, and reagent purge lines. Thirty psi gas pressure is required.

Auxiliary Gas Inlet: Provides oxygen gas flow to POC furnace for oxidation of purgeable organic carbon. Ten psi gas pressure is required. This gas supply is only required if the Purgeables Option is installed (OI Part #164-533).

Actuator Gas Manifold: Manifold block with four (basic unit only), six (process option) or eight (POC option), 12 VDC solenoid valves (OI Part #166-349). Actuation of these values is controlled

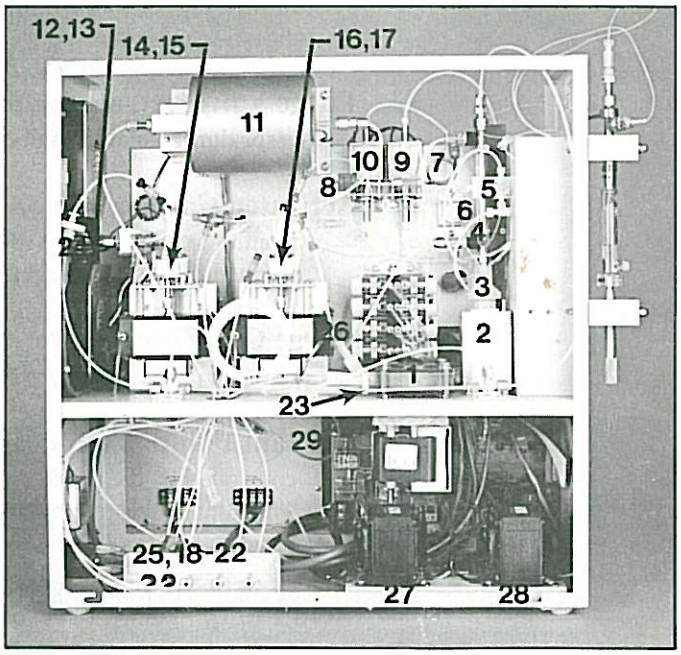


Fig. 2.4 Left Bay

1. *Sample Pump*
2. *Digestion Vessel Heater Block*
3. *Digestion Vessel*
4. *Condensation Chamber*
5. *Injection Port*
6. *Purge Valve*
7. *Activated Carbon Scrubber*
8. *Purge Gas Check Valve*
9. *Oxidant Pump*
10. *Acid Pump*
11. *Furnace*
12. *Primary Trap (Innermost)*
13. *Purgeables Trap (Outermost)*
14. *Primary Trap Valve (Innermost)*
15. *Purgeables Trap Valve (Outermost)*
16. *Drain Valve (Innermost)*
17. *Sample Loop Valve (Outermost)*
18. *Actuator Gas Inlet*
19. *Nitrogen Gas Inlet*
20. *Auxiliary Gas Inlet*
21. *Sample Loop Drain*
22. *Digestion Vessel Drain Line*
23. *Permeation Tube*
24. *Gas Liquid Separator*
25. *External Sample Purge Line*
26. *Actuator Gas Manifold*
27. *POC Trap Transformer*
28. *Primary Trap Transformer*
29. *A.C. Printed Circuit Board*

by the processor for automated switching of valves.

POC Trap Transformer: Power transformer supplying current for heating POC trap. This transformer is part of the Purgeables Option (OI Part #164-533).

Primary Trap Transformer: Power transformer supplying current for heating Primary Trap.

AC Power Control Board: Provides for AC power distribution to heated zones, IR detector and printer. A 15 psi pressure cutoff switch is also located on this board.

Sample Loop Drain: Drain line from the sample loop and pump. Effluent from this line is excess sample which was not captured by the loop for analysis. This line is a part of the Process Sampling Option (OI Part #164-559).

Digestion Vessel Drain Line: Drains sample which has been stripped of TIC and TOC. This line also has an inline one way check valve to prevent external contaminants from entering the instrument.

Permeation Tube: Used for drying the gas stream flowing to the traps. This is a coaxial tube set containing a hydroscopic, ion exchange membrane in a continuous drying process to selectively remove water vapor from mixed gas streams. The membrane is a proprietary extrudible dessicant in tubular form inside an outer tube shell. When an intermittently wet gas stream flows through the inner tube then purges the shell in a counter-current fashion, water vapor molecules are transferred through the walls of the tubing.

Gas-Liquid Separator: An inline membrane filter to prevent the accidental passage of liquid into the infrared detector carrier gas train.

External Sample Purge Line: Provides nitrogen purge gas (ca. 300 ml/min) for external sparging of samples for TIC removal when samples have been pre-acidified.

Infrared Detector - Left Bay View

IR Case Purge/Reagent Bottle Purge: Tee fitting to provide a low volume, inert gas flow into IR detector case to reduce background noise due to ambient CO_2. The 1/16" tubing from either side of the TEE provides inert gas flow to the oxidant and acid reagent bottles to prevent CO_2 contamination (reduce instrument blank).

IR Sample In: 1/8" Swagelok bulkhead union from primary trap valve (or POC furnace if Purgeables Option is installed) into IR detector sample cell.

IR Sample Out: 1/8" Swagelok bulkhead union out of IR detector sample cell leading to permeation tube counter-current gas inlet.

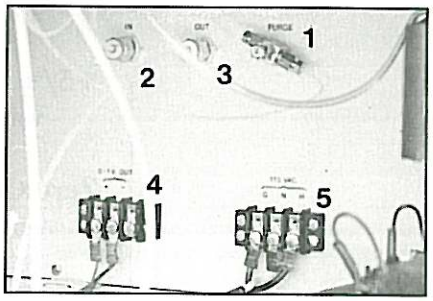


Fig. 2.5 Infrared Detector - Left Bay View
1. *IR Case Purge/Reagent Bottle Purge*
2. *IR Sample In*
3. *IR Sample Out*
4. *0-1 Volt Analog Output Terminal*
5. *IR Power Input Terminal*

0-1 Volt Analog Output Terminal: Provides IR output signal to the I/O board detector signal cable. Wire connections are:

Red: + terminal
Black: - terminal
Green: - terminal

IR Power Input Terminal: Supplies IR detector with 115 VAC power from AC power board. Wire connections are:

Green: G terminal
White: N terminal
Black: H terminal

Right Bay View

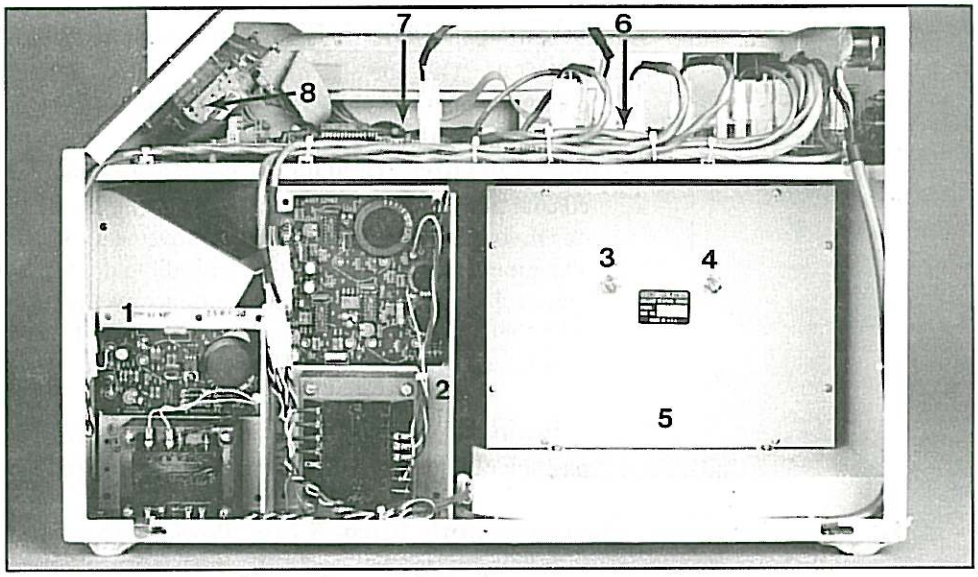


Fig. 2.6 Right Bay View

1. *+12 Volt Power Supply*
2. *+5/± 15 Volt Power Supply*
3. *Span Control*
4. *Zero Control*
5. *Infrared Gas Analyzer*
6. *I/O Board*
7. *Processor Board*
8. *Display Board*
9. *Thermocouple Mux Board*

+12 Volt Power Supply: Provides power to operate all 12 volt DC valves, solenoids, pumps, etc. located in the chemical bay of the instrument.

+5/±15 Volt Power Supply: Provides the regulated + 5 volts DC and ± 15 volts DC required by the digital and analog circuitry.

Zero Control: Used for adjusting the IR offset (baseline) of the instrument while flowing zero gas. Millivolt output of IR may be monitored in IR Output display mode.

Span Control: Factory set gain control. Not to be adjusted except during IR linearization procedure. Contact O.I. Analytical service department if IR gain is suspected as a problem before making any adjustments with this control.

Infrared Gas Analyzer - Right Bay View: Measures mass of carbon in CO_2 samples which are introduced into carrier gas stream by thermal desorption of

the primary trap. This highly selective non-dispersive infrared analyzer (NDIR), incorporates a single-beam photometric system and a detector with a micro-flow sensor. This design, assures high reliability, sensitivity, accuracy and stability.

I/O Board: Provides the input and output interface, signal processing, and analog to digital conversion functions of the microprocessor controller electronics.

Thermocouple MUX Board: See section on Thermocouple MUX Board for a detailed description of MUX board components. Not shown in Fig. 2.6, but seen in left bay housing, when cover is removed, when viewed from the right side.

Processor Board: Main system controller board contains the microprocessor, RAM memory, system firmware and front panel control circuitry.

Display Board: Provides the electrical interface between the processor board and front panel LEDS, alpha-numeric display and switches.

Rear View

Relay #1 Output Terminals (High Alarm Output): Energized when the measured TIC concentration becomes equal to or greater than the TIC Low Limit setting or when the measured TOC concentration decreases below the TOC Low Limit setting. The normally open (N.O.) terminal is shorted to common (COM) when energized.

Main AC Power Receptical: Receives 115 VAC power for operation of the instrument.

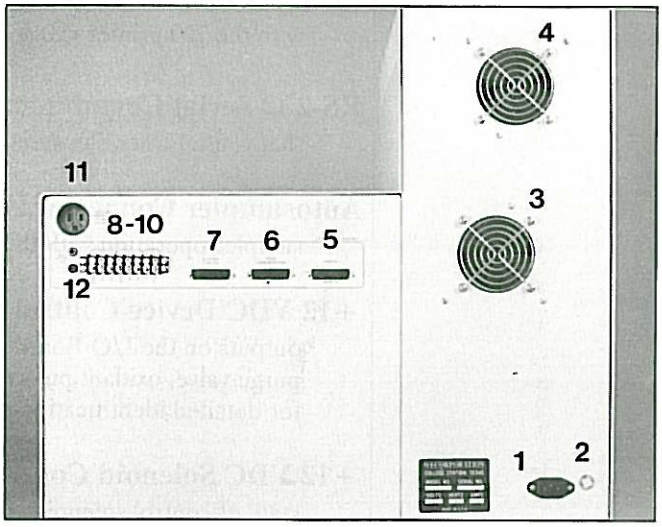


Fig. 2.7 Rear View

1. *Main AC Power Receptical*
2. *Main Power Circuit Breaker*
3. *Primary Trap Fan*
4. *POC Trap Fan*
5. *Parallel Printer Connector*
6. *Serial Communications Connector*
7. *External Control (Auto-sampler) Connector*
8. *Relay #1 Output Terminals (High Alarm Output)*
9. *Relay #2 Output Terminals (Low Alarm Output)*
10. *Auxiliary Terminals*
11. *120 Volt AC Power Outlet*
12. *0-1 Volt Analog Output*

Main Power Circuit Breaker: 10 amp circuit breaker for main power voltage protection.

Primary Trap Fan: Blows ambient air on the primary trap during trap cooling times.

POC Trap Fan: Blows ambient air on the POC trap during trap cooling times.

Parallel Printer Connector: Provides connection to Centronics-compatible parallel interface printer.

Serial Communications Connector: Provides connection to RS-232 serial interface for remote communication.

External Control (Autosampler) Connector: Provides connection to autosampler or other TTL-controlled device.

Relay #2 Output Terminals (Low Alarm Output): Energized when the measured TIC concentration decreases below the TIC Low Limit setting or when the measured TOC concentration decreases below the TOC Low Limit setting. The normally open (N.O.) terminal is shorted to common (COM) when energized.

120 Volt AC Power Outlet: Switched power receptacle for printer.

0-1 Volt Analog Output: Banana plug output for monitoring NDIR with a strip chart recorder.

Auxiliary Terminals: For input of signal from the Model 4420 ELCD when the POX option is installed (OI Part #174-227).

Processor Board And I/O Board

Printer Connector: Supports any Centronics type parallel port printer when used with the 700 printer cable.

RS-232 Serial Communications Connector: A DB-25 type serial connector that is used when the serial port option is installed.

Autosampler Connector: Supplies all of the control signals to synchronize auto sampler operation with the analyzer.

+12 VDC Device Control Indicators: LEDs that show the state of the DC outputs on the I/O Board. They indicate power applied to the spare output, purge valve, oxidant pump and acid pump. See DC Power Control LEDs section for detailed identification of these LEDs.

+12 VDC Solenoid Control Indicators: Four pair of LEDs that show the state of control solenoids that operate the pneumatically controlled 6-port valves. These indicators show the position of the POC trap valve, sample loop valve, sample drain valve and primary trap valve. See DC Power Control LEDs section for detailed identification of these LEDs.

External Control/Alarm Relays: General purpose relays may be configured to signal an alarm condition should the analysis results exceed preset alarm limits. Set points may be set independently for TIC and TOC results. This feature is provided for unattended on-line operation.

Relay Output Connector: Connects to a cable that leads to the rear panel mounted relay terminal block.

Valve Control Solenoid Connector: Connects to a cable that leads to the valve control solenoid bracket. This connector supplies +12 VDC signals that control the position of the 6-port valves.

Acid Pump Connector: Connects to a cable that leads to the acid metering pump.

Oxidant Pump Connector: Connects to a cable that leads to the oxidant metering pump.

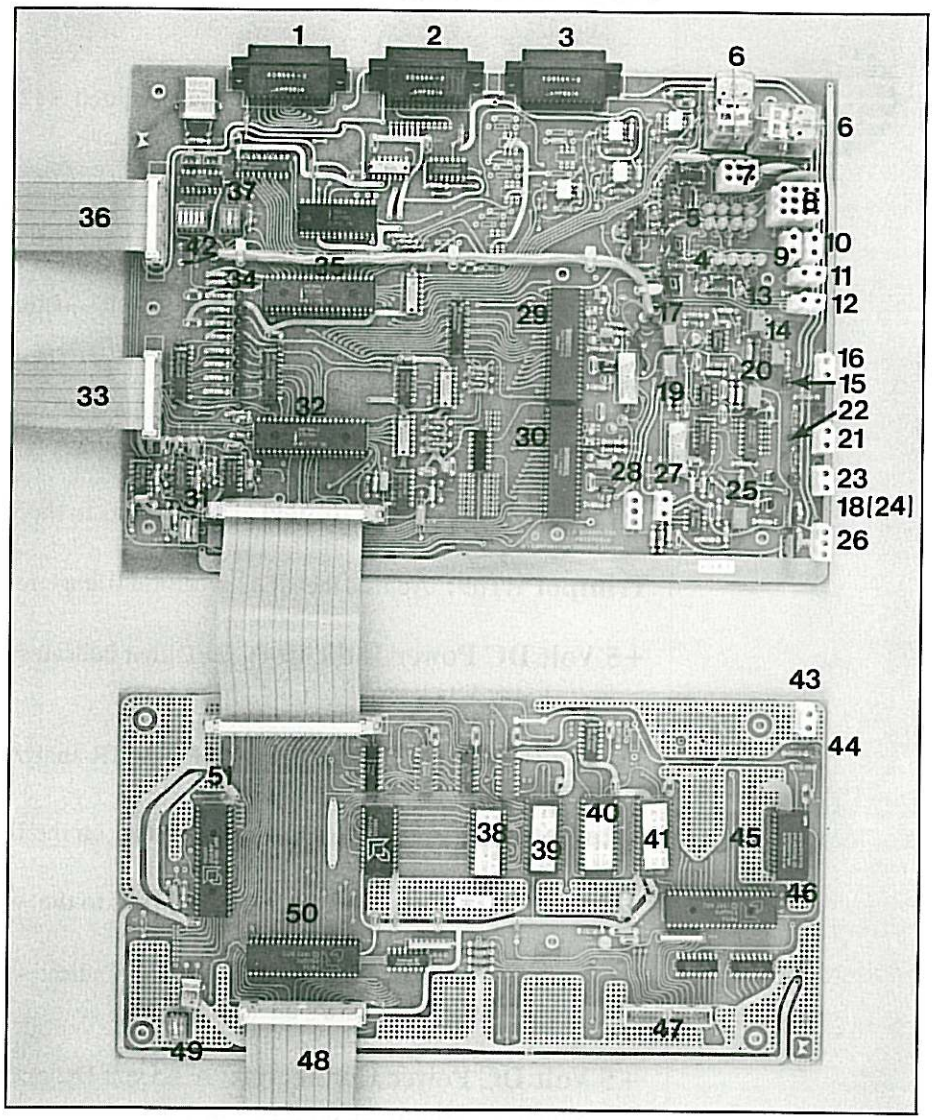


Fig. 2.8 Processor Board and I/O Board

1. *Printer Connector*
2. *RS-232 Serial Communications Connector*
3. *Autosampler Connector*
4. *+12 VDC Device Control Indicators*
5. *+12 VDC Solenoid Control Indicators*
6. *External Control/Alarm Relays*
7. *Relay Output Connector*
8. *Valve Control Solenoid Connector*
9. *Acid Pump Connector*
10. *Oxidant Pump Connnector*
11. *Purge Valve Connector*
12. *Spare DC Control Connector*
13. *Trimpot R114*
14. *Trimpot R111*
15. *+12 Volt DC Power Indicator*
16. *+12 Volt DC Power Connector*
17. *Trimpot R109*
18. *15 Volt DC Power Indicator*
19. *Trimpot R91*
20. *Trimpot R93*
21. *+5 Volt DC Power Connector*
22. *-15 Volt DC Power Indicator*
23. *+5 Volt DC Power Connector*
24. *+15 Volt DC Power Indicator*
25. *Trimpot R101*
26. *+15 Volt DC Power Connector*
27. *IR Recorder Connector*
28. *IR Analyzer Input Connector*
29. *ADC #1*
30. *ADC #2*
31. *Normal/By-pass Switch*
32. *I/O Port I.C.*
33. *AC Power Control Board Ribbon Cable*
34. *AC Power Indicators*
35. *I/O Port I. C.*
36. *Thermocouple MUX Board Ribbon Cable*
37. *50/60 Hertz Switch*
38. *EPROM Socket EP-3*
39. *EPROM Socket EP-0*
40. *EPROM Socket EP-1*
41. *EPROM Socket EP-2*
42. *Baud Rate Dip Switch*
43. *+5 Volt DC Power Connector*
44. *+5 Volt DC Power Indicator*
45. *Ram Memory I.C.*
46. *I/O Port Chip*
47. *Keypad Ribbon Cable*
48. *Display Board Ribbon Cable*
49. *Normal/Test Switch*
50. *I/O Port Chip*
51. *8085 Microprocessor Chip*

Purge Valve Connector: Connects to a cable that leads to the purge valve solenoid.

Spare DC Control Connector: Uncommitted +12 volt control output controllable by system software.

Trimpot R114: Adjusts the accuracy of the temperature control analog to digital converter, ADC #2.

Trimpot R111: Adjusts the accuracy of the IR analyzer analog to digital converter, ADC #1.

+12 Volt DC Power Indicator: LED that indicates the presence of +12 volt power to the I/O board from the power supply.

+12 Volt DC Power Connector: Connects to the +12 volt DC power supply.

Trimpot R109: Adjusts the ADC precision voltage reference.

+5 Volt DC Power Indicator: LED that indicates the presence of + 5 volt power to the I/O board from the power supply.

Trimpot R91: DC offset adjustment for the IR analyzer ADC scaling circuit.

Trimpot R93: Adjusts the 10X scaling factor on the IR + analog signal.

+5 Volt DC Power Connector: Connects to the + 5 volt DC power supply.

-15 Volt DC Power Indicator: LED that indicates the presence of -15 volt power to the I/O board's analog circuitry.

+5 Volt DC Power Connector: A +5 volt DC output that connects to and supplies power to the processor board.

+15 Volt DC Power Indicator: LED that indicates the presence of +15 volt DC power to the analog circuitry of the I/O board.

Trimpot R101: Adjusts the 10X scaling factor on the IR-analog signal.

+15 Volt DC Power Connector: Connects to the +15 volt DC power supply and supplies power to all analog circuitry in the analyzer.

IR Recorder Connector: Connects to the rear panel strip chart recorder connector.

IR Analyzer Input Connector: Differential input that connects to the 0-1 volt analog output of the IR analyzer.

ADC #1: 12 bit analog to digital converter used to convert the IR gas analyzer analog output signal into digital format at a rate of 20 conversions per second.

ADC #2: 12 bit ADC used to digitize the amplified thermocouple signals. This data

is accessed by the temperature control software for control of the four heated zones of the analyzer.

Normal/By-pass Switch: Used to by-pass the safety shutdown circuit of the analyzer that prevents heated device damage if processor control is lost. Leave in the NORMAL position.

I/O Port I.C.: Large scale integration (LSI) port chip that controls the AC and DC devices in the chemical process train.

AC Power Control Board Ribbon Cable: Supplies control signals to the 8 AC power control triac drivers on the AC Power Control Board.

AC Power Indicators: LEDs that indicate the on/off status of each of the eight channels of controlled 120 volt AC power. Flashing LEDs indicate that the power level is being controlled by time proportioning.

I/O Port I.C.: LSI port chip that supplies the printer and autosampler control signals.

Thermocouple MUX Board Ribbon Cable: Carries control signals and power to the T.C. MUX board and analog temperature signals to ADC #2.

50/60 Hertz Switch: Allows selection of 50 or 60 hertz line frequency operation.

EPROM Socket EP-3: Socket for optional program ROM.

EPROM Socket EP-0: Socket for system software EPROM #0, a 2764 8kx8 bit device.

EPROM Socket EP-1: Socket for system software.

EPROM Socket EP-2: Socket for system software.

Baud Rate Dip Switch: User selected switch configuration for serial port communications. (see installation of internal options for Baud Rate Settings)

+5 Volt DC Power Connector: Supplies + 5 volt power from the I/O board to the processor board.

+5 Volt DC Power Indicator: LED that indicates the presence of + 5 volt DC power to the processor board logic circuitry.

RAM Memory I.C.: 2k byte low power CMOS RAM memory chip.

I/O Port Chip: LSI port chip that controls the front panel LED displays and connects to the system keypad.

Keypad Ribbon Cable: Connects to the system keypad.

Display Board Ribbon Cable: Connects to the system control panel and supplies ASCII data and control signals to the alpha-numeric display screen and panel LEDs.

Warning: This Normal/Test switch should ALWAYS be in the NORMAL position when the AC power control board and the heaters are connected for operation.

Normal/Test Switch: This switch allows testing of the processor board and I/O board without the AC power control board.

I/O Port Chip: LSI port chip that provides alpha-numeric display control signals and matrix switch scan signals to the keypad.

8085 Microprocessor Chip: The central processor unit (CPU) for all analyzer functions. The CPU operates at a rate of 2.6 million instruction cycles per second.

DC Power Control LEDs

These LEDs, located on the I/O board, indicate the position of the +12 volt DC operated devices in the chemical process train.

Purge Valve: The purge gas is flowing when the LED is illuminated.

Spare DC Output, software controlled.

Acid Metering Pump: Each flash of the LED indicates one 100 ul stroke of the acid pump.

Oxidant Metering Pump: Each flash indicates one 100 ul stroke of the oxidant pump.

POC Trap Valve Position (inactive)

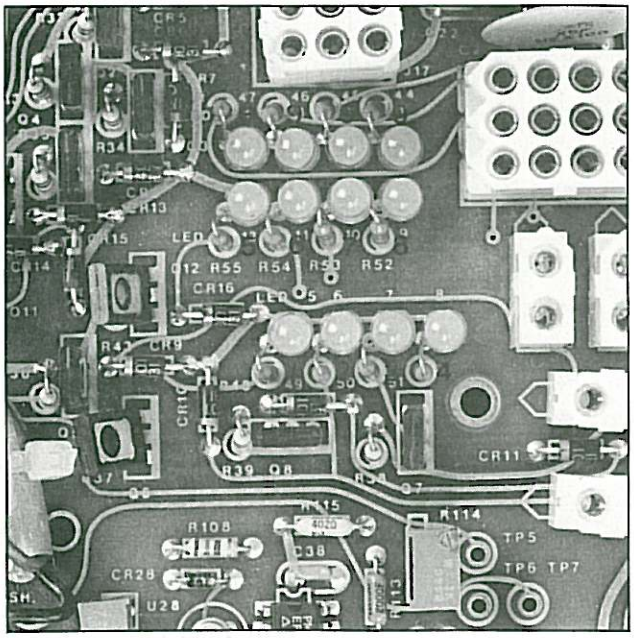


Fig. 2.9 DC Power Control LEDs

1. *Purge Valve*
2. *Spare DC Output*
3. *Acid Metering Pump*
4. *Oxidant Metering Pump*
5. *POC Trap Valve Position*
6. *Sample Loop Valve Position*
7. *Sample Drain Valve Position*
8. *Primary Trap Valve Postion*
9. *Indicates the active position of 5-8 above.*

Sample Loop Valve Position (inactive)

Sample Drain Valve Position (inactive)

Primary Trap Valve Position (inactive)

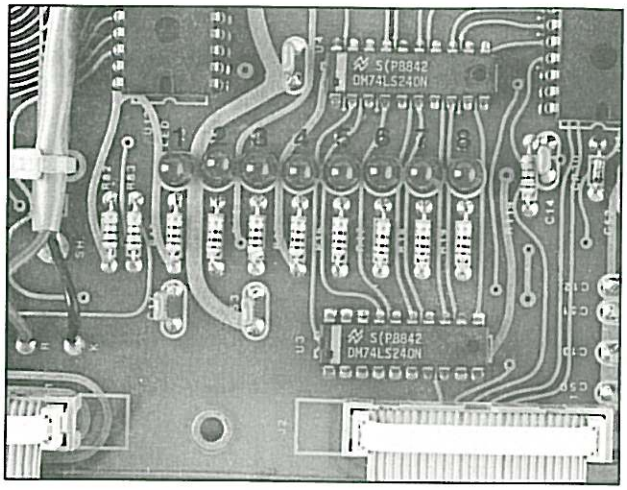
AC Power Control LEDs

115 VAC power is being supplied to these components when the corresponding LED is illuminated:

Primary Trap Power
POC Trap Power
Digestion Vessel Power
POC Furnace Power
Primary Trap Fan Power
POC Trap Fan Power
Sample Pump Power
Spare AC Power

Fig. 2.10 AC Power Control LEDs

1. *Primary Trap Power*
2. *POC Trap Power*
3. *Digestion Vessel Power*
4. *POC Furnace Power*
5. *Primary Trap Fan Power*
6. *POC Trap Fan Power*
7. *Sample Pump Power*
8. *Spare AC Power*



Thermocouple MUX Board

Scans the primary trap, POC-trap, digestion block, and the POC furnace temperatures through the thermocouple connections. Each temperature is checked four times a second.

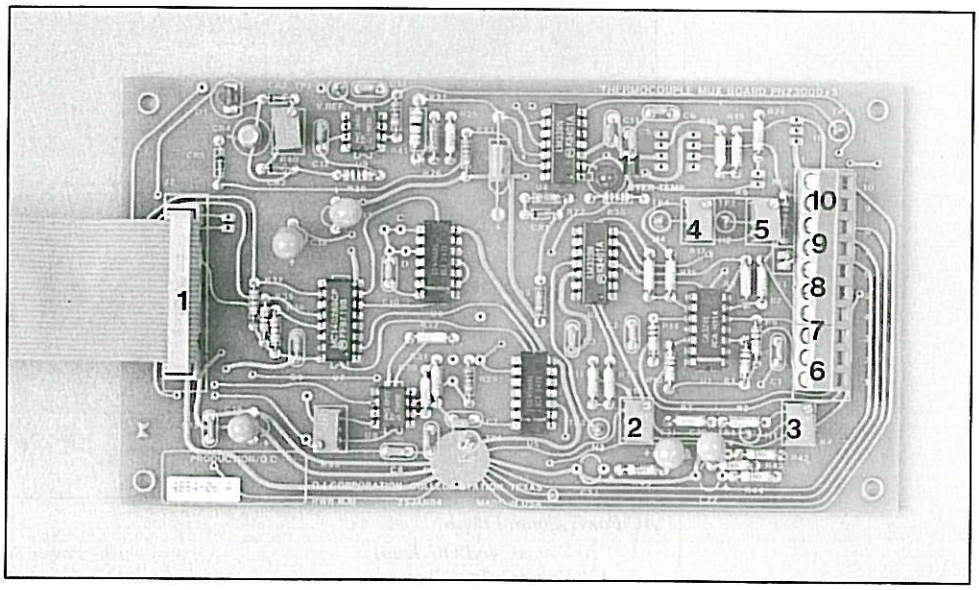


Fig. 2.11 Thermocouple MUX Board

1. *Ribbon Cable Connector*
2. *Digestion Block Overtemperature Set point Adjustment*
3. *Primary Trap Overtemperature Set point Adjustment*
4. *POC Furnace Overtemperature Set point Adjustment*
5. *POC Trap Overtemperature Set point Adjustment*
6. *Primary Trap Themocouple Terminals*
7. *POC Trap Thermocouple Terminals*
8. *Digestion Block Thermocoule Terminals*
9. *POC Furnace Thermocouple Terminals*
10. *Spare Terminals*
11. *Overtemperature LED*

Ribbon Cable Connector: Connects to ribbon cable that leads to the I/O board.

Overtemp Set Point Adjustment: Adjusts the maximum operating temperature of the digestion block heater before an overtemperature safety shutdown occurs.

Digestion Block Overtemp Set Point Adjustments

- Primary Trap Overtemperature Set Point

- POC Furnace Overtemperature Set Point
- POC Trap Overtemperature Set Point

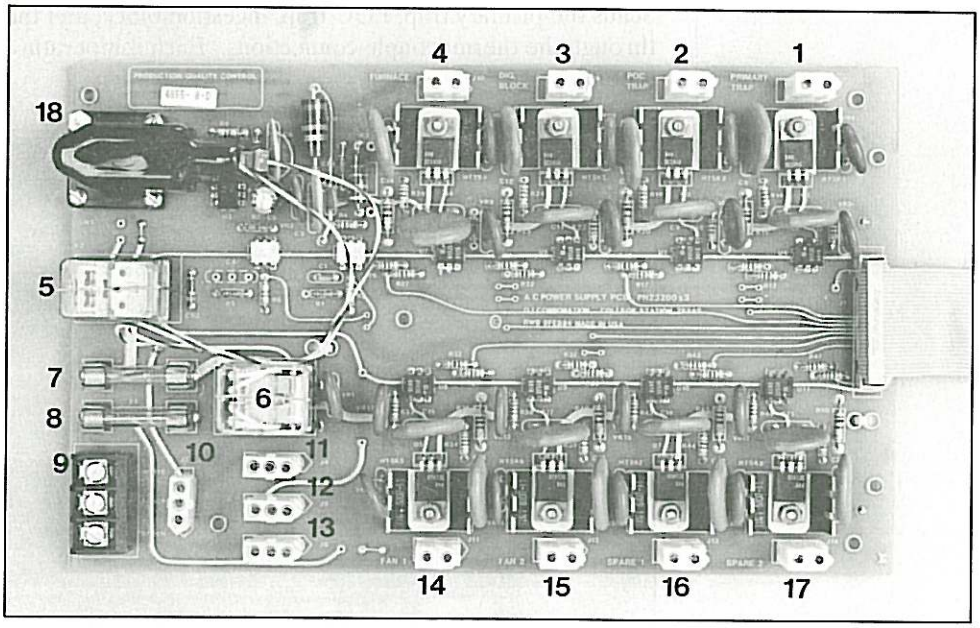
Terminals

- Primary Trap Thermocouple Terminals
- POC Trap Thermocouple Terminals
- Digestion Block Thermocouple Terminals
- POC Furnace Thermocouple Terminals

Overtemperature LED: Illuminates when over temperature safety shutdown of any heated zone occurs.

AC Power Control Board

Controls the 115 volt primary power to the DC power supplies, the IR detector, the external printer power connector, and the on-board triac switch for the heaters.



AC Power Control Board

1. *120 Volt AC (NPOC Trap)*
2. *120 Volt AC (POC Trap)*
3. *120 Volt AC (Digestion Block)*
4. *120 Volt AC (POC Furnace)*
5. *Heater Overtemperature Protection Relay*
6. *Main AC Power Control Relay*
7. *Fuse, 6 Amp*
8. *Fuse, 4 Amp*
9. *Main Power Terminal Block*
10. *Power Switch Connector*
11. *Switched AC Connector, J4*
12. *Swithed AC Connector, J5*
13. *Switched AC Connector, J6*
14. *120 Volt AC (NPOC Fan)*
15. *120 Volt AC (POC Fan)*
16. *120 Volt AC (Sample Pump)*
17. *120 Volt AC (Spare)*
18. *Pressure Cutoff Switch*

120 Volt AC: Output to primary trap transformer.

120 Volt AC: Output to POC trap transformer.

120 Volt AC: Output to Digestion block heater.

120 Volt AC: Output to POC furnace heater.

Heater Overtemperature Protection Relay: Disconnects power if overheating occurs on any heated zone.

Main AC Power Control Relay: Controls main supply power to the analyzer and printer.

Fuse, 6 Amp: Protects the four 120 volt AC heater control outputs.

Fuse, 4 Amp: Protects the remaining four 120 volt AC control outputs.

Main Power Terminal Block: Supplies 120 volt AC power to the AC power control board.

Power Switch Connector: Connects to the front panel power switch.

Switched AC Connector, J4: Supplies 120 volt AC power to the regulated DC power supplies.

Switched AC Connector, J5: Supplies 120 volt AC power to the IR analyzer.

Switched AC Connector, J6: Supplies 120 volt AC power to the printer power connector on the instrument rear panel.

120 Volt AC: Output to the primary trap fan.

120 Volt AC: Output to the POC trap fan.

120 Volt AC: Output to the Sample Pump.

Spare 120 volt AC output, software controlled.

Pressure cutoff switch. : If inlet gas pressure drops below 15 psi this switch interrupts all AC power to instrument.

Chapter 3
Installation

In **Chapter 2** the names and functions of the various components of the 700 were outlined. Names of components were printed in **boldface type** to highlight them. These names are used here to refer to components involved in the installation of the instrument.

This chapter deals with the stepwise procedures used for properly installing the 700 and its optional autosampler. The chapter begins with some general information including safety aspects, then discusses materials needed for installation which are not included with the basic instrument. You should gather the materials outlined here before attempting the installation, then proceed in a stepwise fashion through the instructions beginning with Initial Setup.

General Information

Internal vs. External Options

The instrument is shipped with any external options packed in separate boxes and any internal options already installed within the Model 700. Options which can be installed internal or external to the Model 700 are as follows:

Internal

- Serial Communications Port (OI Part #169-070)
- Process Sampling Capability (OI Part #164-559)
- Purgeable Organic Carbon Capability (OI Part #164-533)

External

- Voltage Convertor, 230 VAC to 115 VAC (OI Part #169-179)
- Voltage Convertor, 100 VAC to 115 VAC (OI Part #169-187)
- Strip Chart Recorder (OI Part #138-596)
- Printer (OI Part #169-004)
- Multiplexed Process Sampling Capability (OI Part #169-088)
- Autosampling Capability (OI Part #169-012)
- Ampule (Solids) Capability (OI Part #164-541)
- Wafertoc Capability (OI Part #169-096)
- Purgeable Organic Halide (OI Part #174-227)

Removing Instrument Covers

Components mounted inside the left and right bays are described in previous sections. These components may be exposed by removing the left and right bay covers. Cover removal is also necessary for installation of some options. Covers are removed in the following manner:

- Remove shipping screws located near the bottom of the covers on the left and right sides of the instrument.

- Force each cover forward 0.5" (1 cm) then lift upward to remove.

Safety

- The exposure to personal hazards for this instrument and the methodology employed has not been precisely defined. The instructions for installation and operation given in this manual are believed to be a thorough account for proper and safe operation. However, it is the responsibility of each laboratory for maintaining the instrument in a condition suitable for safe use. Guidelines for maintenance are given later in this manual.

- The toxicity or potential health risk hazard of chemicals used in this method has not been precisely defined. However, all chemicals, and samples used should be treated as a potential health risk, and exposure to the materials should be minimized. Each laboratory is responsible for maintaining awarness of OSHA regulations regarding safe handling of chemicals and associated equipment used in this method.

- Phosphoric acid has been identified as a corrosive and toxic material. Pure material and diluted solutions of this compound should be handled in a manner consistent with OSHA regulations. Appropriate skin and eye protection should be worn when the analyst handles any materials containing this substance.

- The salts of peroxydisulfate ($Na_2S_2O_8$ and others) and solutions containing these salts have been identified as strong oxidizers, corrosive and toxic materials. Pure materials and diluted solutions of these compounds should be handled in a manner consistant with OSHA regulations. Appropriate skin and eye protection should be worn when the analyst handles any materials containing these salts. Caution should be exercised when handling these salts, or solutions, containing these salts in the presence of organic materials which could result in accidental contact.

- Potassium Biphthalate and Sodium Carbonate have been identified as chemical irritants to human skin and eyes. Pure materials and stock solutions of these compounds should be handled in a manner consistent with OSHA regulations. Appropriate skin and eye protection should be worn when the analyst handles any materials containing these substances. The analyst should avoid exposure to fume or dust inhalation.

- Nitrogen and helium have been identified as asphyxiants. These gases and their compressed cylinders should be handled and stored in a manner consistent with OSHA regulations. Adequate ventilation should be maintained in areas where these materials are used and stored. The analyst should avoid prolonged exposure to high concentrations of these gasses.

- Oxygen has been identified as an oxidizer. This gas and compressed cylinder containing this gas should be handled and stored in a manner consistent with OSHA regulations. Open flames and easily ignited materials should not be brought in contact with the pure gas except under approved, controlled conditions by the analyst. The analyst should also avoid prolonged exposure to high concentrations of this gas.

Installation of Basic Unit

Materials Needed Before Installation

Before installation of the Model 700 can begin, there are several items the user must

have on hand. In the list that follows are the bare necessities for bringing the instrument into operation. The user will of course find with experience, and depending on the options installed, that there are other items that are useful to have on hand in their particular laboratory.

- Source of high purity reagent water, TOC 200 ppb C or less (OI Part #169-301).
- 85% Ortho-phosphoric acid, ACS reagent grade (OI Part #110-080 (500 ml)).
- Sodium persulfate crystals, ACS reagent grade (OI Part #178-848 (500 grams)).
- Potassium biphthalate, primary standard crystals and/or 1000 ppm C standard solution (OI Part #136-954 for crystals and #169-252 for 10 ml of standard).
- Sodium carbonate, anhydrous powder and/or 1000 ppm C standard solution (OI Part #136-462 for the anhydrous powder and #169-244 for 10 ml of standard).
- Two stage nitrogen or helium regulator (0-60 psig) (OI Part #155-417 (specify N_2 or He when ordering)). If the POC option is installed, then a two stage oxygen regulator is necessary also (OI Part #150-326).
- Nitrogen or helium with 99.98% purity or better.

The last 4 items listed here are provided when factory installation (OI Part #141-331) is to be performed.

- 1/8" OD x 0.063" ID gas tubing. OI Part #147-901 is 1/8" OD x 0.063" ID TFE teflon tubing (specify length needed). Twenty feet of this Teflon tubing is suggested, since spare tubing can be used for making sample loops and/or making repairs.
- 1/8"OD x 0.063" ID Swagelok tee fitting (if actuators and carrier/purge gas are to be run from the same gas source) (OI Part #124-750).
- One 1/4" MNPT x 1/8" Swagelok adaptor for adapting most gas regulators to 1/8" OD gas tubing (OI Part #152-215).
- Teflon pipe tape (for pipe threads only) (OI Part #111-005).

The basic Model 700 has a start-up kit. Some options also have their own start-up kits.

Gas and Fluid Connections

- After opening the instrument crate, remove the start-up kits and inspect for completeness. Listings of components are included in the kits.
- Remove the instrument from the case and locate near a suitable gas source and electrical power. Length of the power cord is 180 cm (72 in).
- Connect the 2 gas lines (nitrogen and actuator gas) to the gas inlet bulk heads as shown. Actuator gas may be from a separate cylinder (air, nitrogen, or any other non-flammable gas) or may be provided to the inlet using a tee from the nitrogen gas line.
- Grasp the tube marked SPL DRAIN and pull it outward until it extends from the case at least 30 cm (12 inches).

- Route the drain tube to a waste bottle or other receptacle.

- Turn on the nitrogen gas flow and immediately adjust regulator pressure to 30 psi and check for supply gas leaks with Snoop or other suitable leak detector.

- Fill the reagent bottles with reagents prepared according to instructions in **Chapter 1**.

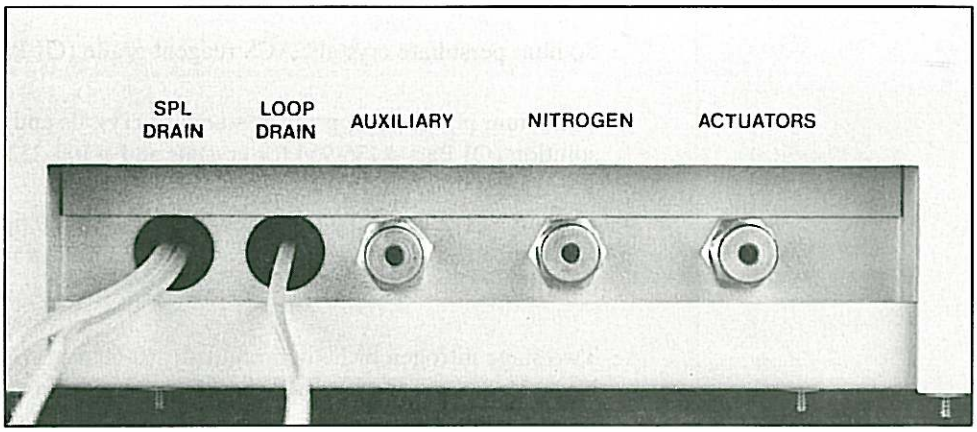


Fig. 3.1 Gas and Fluid Connections

- Match the acid bottle with the reagent tube marked H_3PO_4 (Phosphoric Acid) and the oxidant bottle with the reagent tube marked $Na_2S_2O_8$ (Sodium Persulfate). Insert the corresponding tubes in the bottles, but do not tighten the caps.

CAUTION: If the reagent bottles are not vented, pressure from the bottled purge gas may force reagents into the system and cause damage to some components.

- Confirm that solutions in reagent bottles are being purged.

- Confirm that the septum and cap assembly attached to each reagent tube has a short vent tube protruding through the septum.

- Screw the two reagent bottle caps onto the corresponding reagent bottles and gently tighten.

- Connect any external alarm hardware to the connectors marked RELAY 1 and RELAY 2 according to the convention described in **Chapter 2**.

The Model 700 is designed for operation with 110-125 VAC, 49.5-60.5 Hz power. A voltage convertor should be used for operation with power outside this range (see Chapter 1 for voltage convertor options).

- Connect the power cord to the corresponding receptacle in the rear of the instrument and plug into a source of 115 VAC power.

- Install any optional equipment as described later in this chapter before initial power up.

Initial Power Up

- Turn on the power using the main POWER switch. The following message will appear on the display screen:

- Confirm that the purge and carrier nitrogen gas flow meters show the flow settings specified in **Chapter 1**.

```
TIC/TOC ANALYSIS
00:00:00  STANDBY  <
```

- The screen will display the STANDBY message until the digestion vessel is

heated to the proper temperature. When the digestion vessel reaches its preset temperature, the following message will appear on the display screen:

This heating period is less than 10 minutes. If the RUN/STOP key is pressed during this period, the < in the STANDBY message will change to > and the instrument will wait until the preset digestion vessel temperature has been reached; then the analysis sequence will begin automatically.

```
TIC/TOC ANALYSIS
00:00:00 *READY* <
```

Priming Reagents

Note: Press the CLEAR key so that reagents are carried to drain as they are dispensed.

- Open the keypad drawer and press the PRIME ACID key 20 to 30 times to prime the acid pump.
- Press the **PRIME OXIDANT** key 20 to 30 times to prime the oxidant pump.

Installation of Internal Options

Serial Communications Port

Attach user-provided cable from remote terminal or computer to appropriate connector on the rear of the Model 700.

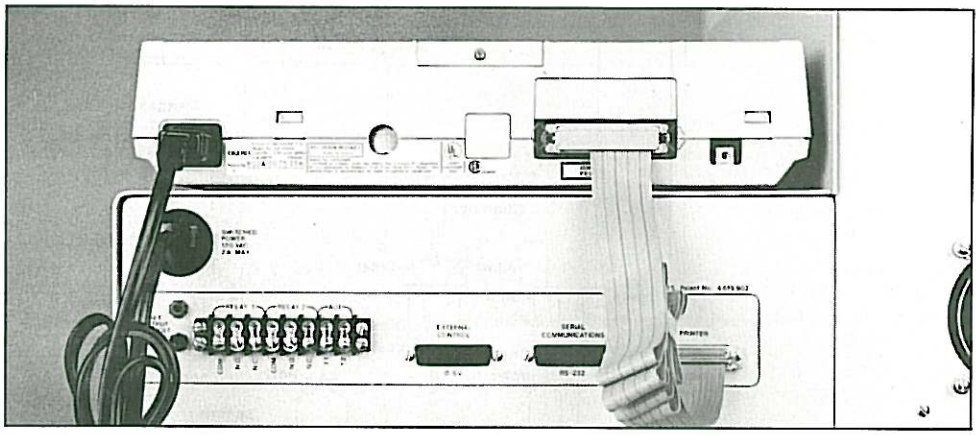


Fig. 3.2 Printer Connections

- Remove the right bay cover and locate the Serial Port Baud Rate Select DIP Switches.
- Select the desired Baud Rate by setting DIP switches 2 through 5 according to the following table: (H means depress the switch-end nearest the word OPEN).

Baud Rate	Switch Number			
	2	3	4	5
110	H	H	H	H
150	H	H	H	L
200	L	H	L	H
300	H	H	L	H
600	L	H	H	L
1200	H	L	H	H
1800	H	L	H	L
2400	L	H	H	H
4800	H	L	L	H
9600	H	L	L	L

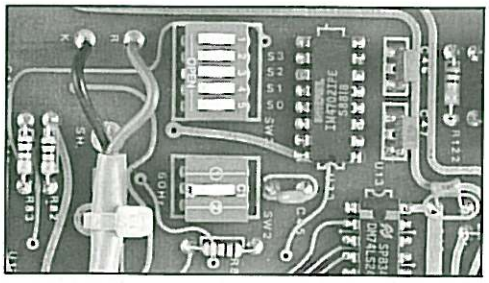


Fig. 3.3 Serial Port-Baud Rate Switch

Process Sampling Option

Press these two inter-connecting Luer-Lok fittings tightly together to avoid air or sample leakage.

Refer to Fig. 3.4 (plumbing schematic) and perform the following steps.

- Grasp the tube marked LOOP DRAIN and pull it outward until it extends from the case at least 12" (30 cm).
- Route this drain tube to a waste bottle or other receptacle.
- Remove teflon sampling line with Luer-Lok fitting from the start-up kit and connect to the loop sampling inlet port.
- Remove the left bay cover, as described earlier in this chapter, and install a sample loop.
- Press the **SET SAMPLE VOLUME** key and enter the volume of the sample loop as given on the tag attached.
- Press **ENTER** to store this volume and automatically set the sample pump run time.

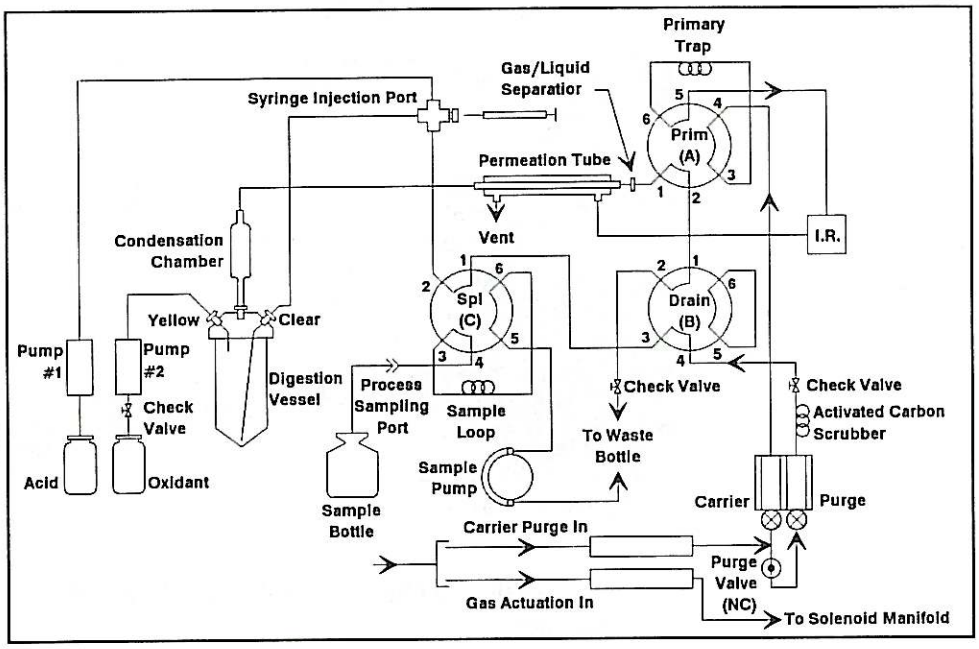


Fig. 3.4 Basic Unit with Process Option Plumbing Schematic

Purgeable Organic Carbon Capability

Refer to Fig. 3.5 (plumbing schematic) and perform the following steps.

- Remove the left bay cover as described earlier in this chapter.
- Remove the quartz furnace tube (pre-packed with catalyst) from the start-up kit and slide through the hole in front of the furnace.
- Fully insert the end of the quartz furnace tube into the corresponding fitting with teflon ferrule set.

This fitting is mounted on a bracket toward the rear of the unit.

CAUTION: Oxygen greatly accelerates combustion. Always check that all oxygen connections are leak tight.

- Insure that the furnace tube passes through the ferrule set and contacts the internal bottom of the mounted fitting.
- Carefully tighten the fitting nut to form a leak-tight seal between the furnace tube and the teflon ferrule set.
- Connect the 1/4" x 1/8" tube union to the open end of the furnace tube.
- Connect an oxygen gas line to the auxiliary gas inlet bulkhead.
- Turn on the oxygen gas flow and immediately adjust regulator pressure to 10 psi. (ca. 30 ml/min as measured at the O_2 inlet tee).

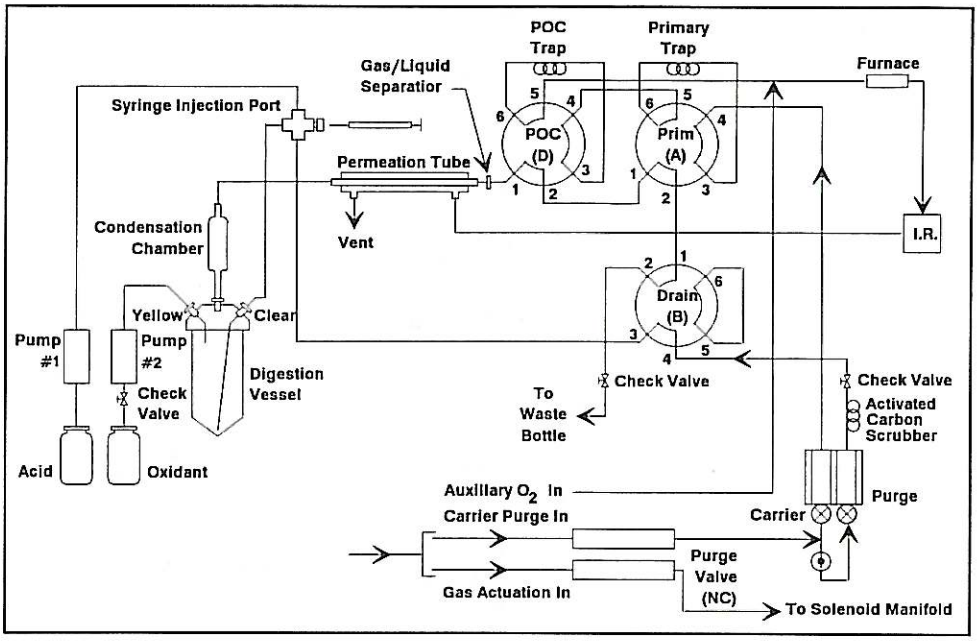


Fig. 3.5 Basic Unit with POC Option Plumbing Schematic

Installation of External Options

Voltage Convertor, 230 VAC to 115 VAC

Converted power is provided on the rear of the Model 700 for the printer. Alternatively, the printer can be plugged into a convertor box outlet.

- Plug the Model 700 power cord into one of the four outlets on the voltage convertor box.
- If any of the following components are to be operated, plug each into a remaining outlet on the voltage convertor box: autosampler, strip chart recorder, ampule purging and sealing unit, Wafertoc heater block.
- Plug the power inlet to the convertor box into a source of 230 VAC.
- Turn on power to convertor box outlets by putting the power switch in the UP position.

Voltage Convertor, 100 VAC to 115 VAC

- Plug the Model 700 power cord into one of the four outlets on the voltage convertor box.

- If any of the following components are to be operated, plug each into a remaining outlet on the voltage convertor box: autosampler, strip chart recorder, ampule purging and sealing unit, Wafertoc heater block.

- Plug the power inlet to the convertor box into a source of 100 VAC.

- Turn on power to convertor box outlets by putting the power switch in the UP position.

Strip Chart Recorder

- Follow instructions in strip chart recorder manual for proper installation of paper, pen, and recorder input leads.

- Plug recorder input leads into the corresponding red and black banana jacks on the rear of the Model 700.

- Plug recorder power cord into 115 VAC outlet and turn on power.

- Zero the recorder if necessary (see recorder manual for instructions).

- Select 1.0 V full scale on the recorder and a chart speed of about 15 cm/hr.

Printer

- Follow instructions in printer manual for proper installation of paper, paper guide and cable.

- Plug the printer into connector labelled PRINTER on the rear of the instrument using the printer interface cable.

- Plug the printer power cord into the SWITCHED POWER outlet as shown in Fig 3.2.

- Turn on printer power.

Use instructions describing Sampler with Wash Station Assembly and Septum Piercing Accessory for units with Wash Station in the Autosampler Instruction Manual.

Autosampler

- Follow instructions in section 2 of the autosampler manual for proper assembly of autosampler and optional septum piercing accessory.

- Place sample racks on autosampler platform boats per instructions in autosampler manual.

- Plug one end of autosampler cable into the matching connector on the rear of the autosampler and the other cable end into the corresponding connector on the rear of the Model 700.

- Plug autosampler power cord into autosampler and into a source of 115 VAC power.

The autosampler has a rear power switch. When power is connected and this switch is in the on position the autosampler will advance forward until the first sample rack is in

the sample fill location at the back of the autosampler platform.

- Connect one end of the loop sampling tube to the loop sampling inlet port and the other end to the autosampler arm as described in the Autosampler Instruction Manual.

- All signals for autosampler control are sent from the Model 700 when the Autosample option is enabled.

Two sizes of teflon tubing guides are provided for use with 1/8 "and 1/16" OD loop sampling tubes.

CAUTION: The sample arm spring (internal to the ASM) is rated at 4 lbs of tension. Always have the hold down foot in place and keep hands and clothing clear of sampling arm when autosampler is in operation.

Water samples cannot be analyzed except by using ampule analysis procedures when the system is plumbed in this manner for ampule analysis. Reverse these steps to regain the plumbing configuration for water sample analysis.

Ampule (Solids) Capability Setup

The Ampule Capability is comprised of two components: (1) an Ampule Purging and Sealing Unit (OI #132-894), which is a free-standing piece of equipment, and (2) an Ampule Breaking Assembly (OI #169-210) which is mounted on and plumbed to the Model 700. The Ampule Purging and Sealing Unit does not need to be located near the Model 700.

Procedures for installation of the Ampule Purging and Sealing Unit and for preparation of samples in ampules are described in the P&S Operator's Manual.

Refer to Fig. 3.6 (plumbing schematic) and Fig. 3.7 (Ampule Capability Setup) and perform the following steps.

- Locate the disassembled Ampule Breaking Assembly and remove the left bay cover, as described earlier in this chapter, for installation.

- Turn off gas flow and power to the Model 700.

- Install the breaking assembly onto the front of the Model 700 as shown.

- Refer to **Chapter 2** for proper identification of components.

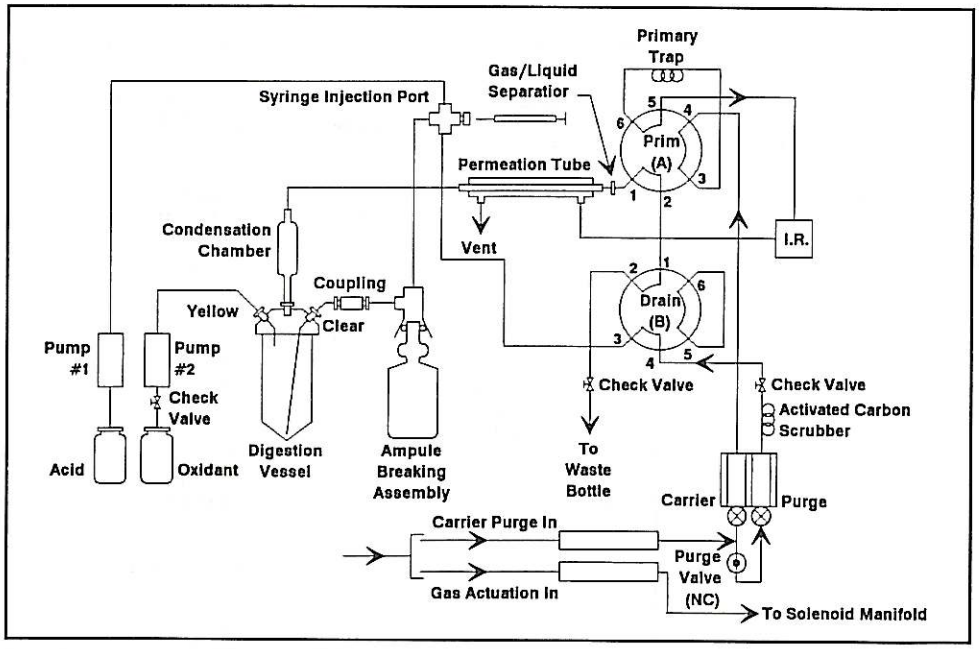


Fig. 3.6 Basic Unit with Ampule Option Plumbing Schematic

Be careful not to pull on tube from digestion vessel.

- Disconnect the tube end fitting from Port 2 of the cross coupling (injection port).
- Connect the tube end fitting from the side arm of the Ampule Breaking Assembly to the tube end fitting from the digestion vessel using the coupling provided. Tighten fingertight.
- Connect the tube end fitting from the purge tube of the Ampule Breaking Assembly to Port 2 of the cross coupling (injection port). Tighten finger-tight.
- Turn on gas flow and power to the Model 700.

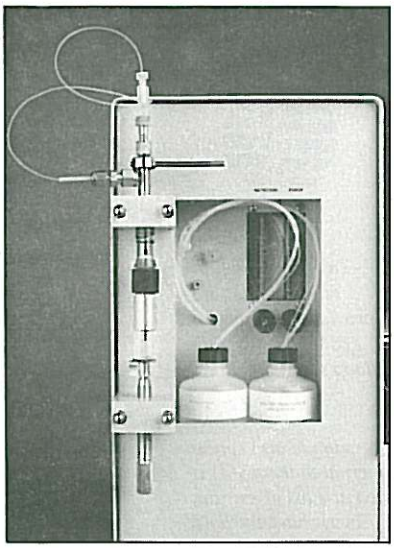


Fig. 3.7 Ampule (Solids) Capability Setup

Wafertoc

Refer to Fig. 3.8 (plumbing schematic) and perform the following steps to interface the wafer digestion chamber with the Model 700.

- Turn off gas flow and power to the Model 700.
- Disconnect Valve A Port 2 (red) from Valve B Port 1 (black). Save the coupling.
- Disconnect Valve B Port 6 (blue) and Valve B port 5 (green). Save the coupling.
- Disconnect Valve B Port 2 (red) from the drain line (red). Save the coupling.
- Connect Valve A Port 2 (red) with Valve B Port 5 (green) using a coupling. Tighten finger-tight.

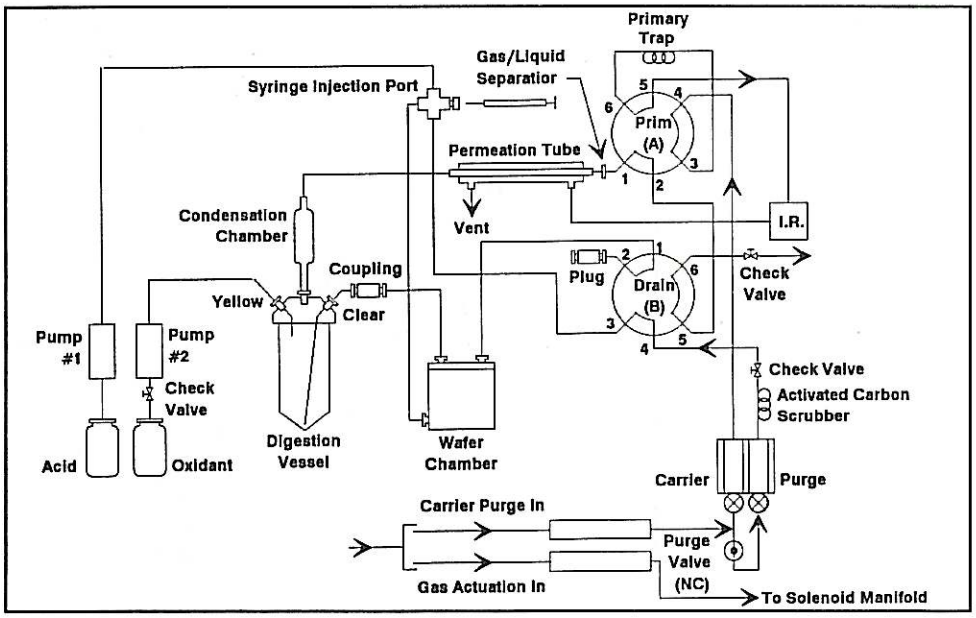


Fig. 3.8 Basic Unit with Wafer TOC Option

- Connect Valve B Port 6 (blue) to the drain line (red) using a coupling. Tighten finger-tight.
- Plug Valve B Port 2 (red) using the plug fitting provided and a coupling. Tighten finger-tight.
- Connect Valve B Port 1 (black) to one of the wafer chamber top tubes using a coupling. Tighten finger-tight.
- Disconnect the tube end fitting from Port 2 of the cross coupling (injection port) and connect it to the other wafer chamber top tube using a coupling. Tighten finger-tight.
- Connect the remaining wafer chamber top tube to the digestion vessel sample drain line with a polypropylene coupling. Tighten finger-tight.
- Turn on gas flow and power to the Model 700.
- Refer to **Chapter 4** for operation.

Chapter 4
Operation

In the last chapter, installation of the 700, its optional autosampler, and other options was described. This chapter deals with the operation of the equipment for analyzing samples. The instructions here assume that the procedures outlined in **Chapter 3, Installation** have been completed. This chapter begins with an overview of the methods used by the 700 for sample analysis, including diagrams of the flow paths during an analysis cycle. It then gives step-by-step instructions for power up, sample introduction, running blanks, calibrating, and running various types of samples.

Overview

A water sample may be introduced into the instrument either by syringe injection, or by means of a calibrated sample loop. The sample loop affords greater consistency of injection volume, whereas the syringe injection port allows injection of microliter quantities of samples of extremely high carbon concentration. Once the sample has been introduced, the **RUN/STOP** key is pressed and the entire analysis sequence is automatic.

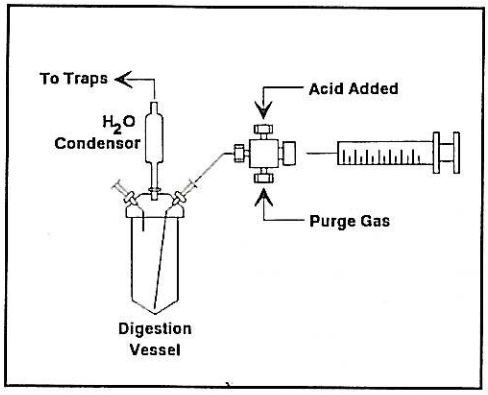


Fig. 4.1 Sample Introduction by Syringe

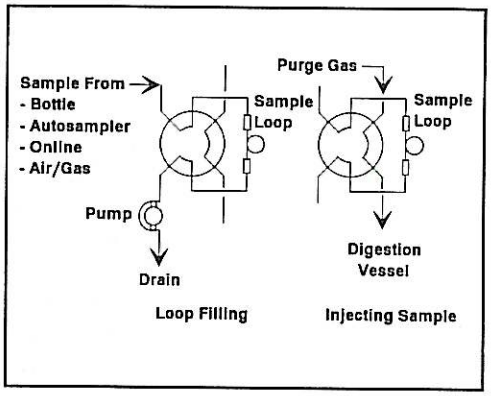


Fig. 4.2 Sample Introduction by Sample Loop

Sample introduction is followed by a metered amount of acid into the digestion vessel.

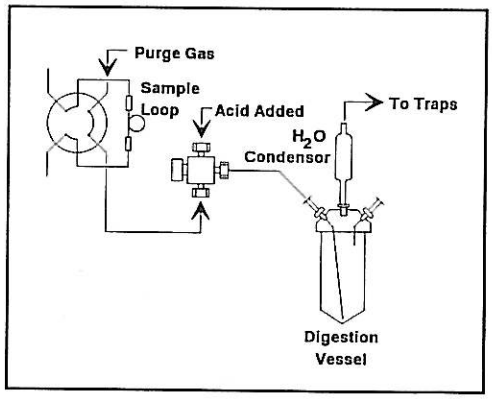


Fig. 4.3 Acid Addition Step

After acid has been added, a gas stream purges out any carbon dioxide formed from inorganic carbon in the sample. This carbon dioxide is carried to a molecular sieve trap held at 25°C where it is trapped and concentrated.

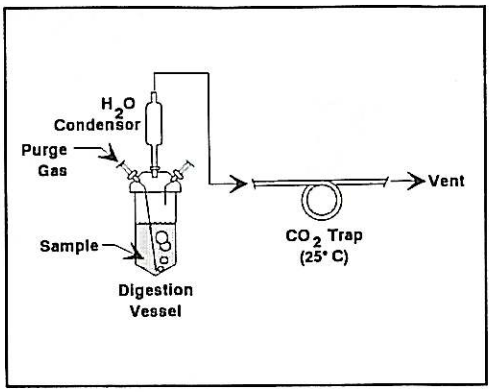


Fig. 4.4 Purge & Trap Step

When purging and trapping is complete, the trap is placed in-line with the infrared detector and rapidly heated to 200°C. A stream of gas desorbs the carbon dioxide from the trap and carries it into the detector. The detector response represents the amount of TIC in the sample. Concentration of TIC displayed may be automatically printed.

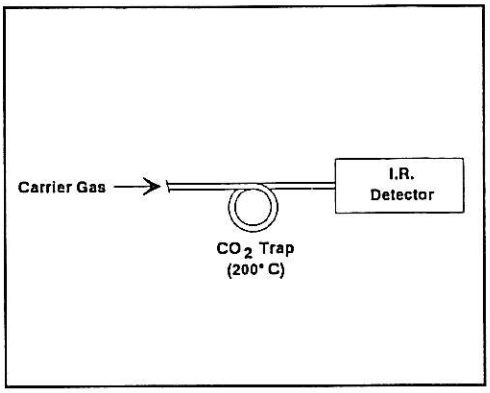


Fig. 4.5 Desorption & Detection

While the carbon dioxide from TIC is being detected, a metered amount of sodium persulfate is added to the sample, which by now has been heated to 100°C. Meanwhile, purge gas flow to the digestion vessel has been shut off. The persulfate reacts with organic carbon in the sample to produce carbon dioxide, which accumulates in the digestion vessel.

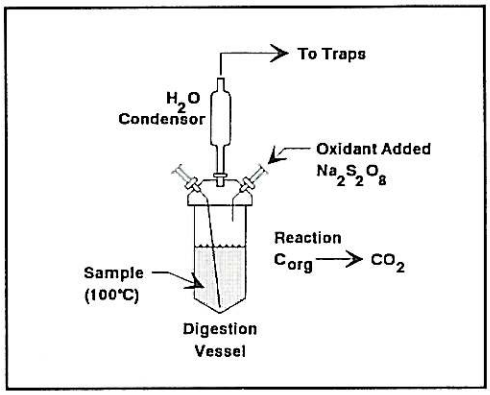


Fig. 4.6 Oxidation Step

After a preset reaction time, the digestion vessel is replaced in-line with the trap and a gas stream purges out any carbon dioxide produced by the persulfate oxidation. This carbon dioxide is carried to the trap held at 25°C where it is trapped and concentrated.

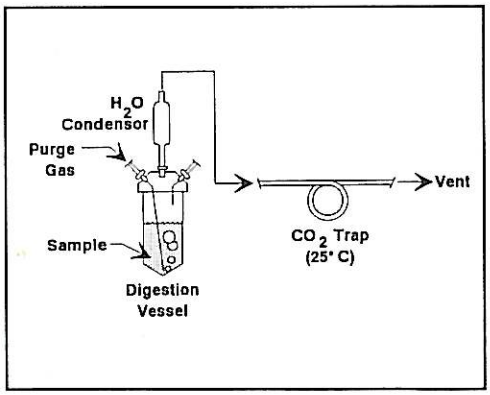


Fig. 4.7 Purge & Trap Step

As before, the trap is then placed in-line with the detector and heated. The carbon dioxide is carried into the detector, and the resulting concentration of TOC in the sample is displayed/printed.

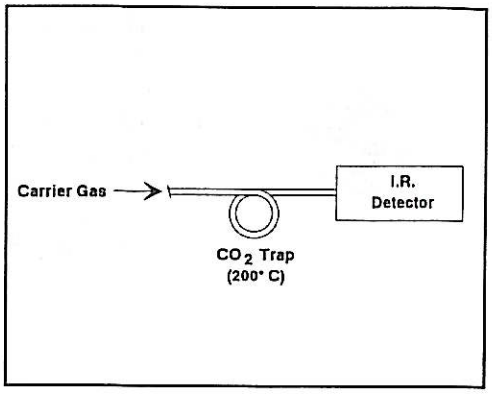


Fig. 4.8 Desorption/Detection

While the carbon dioxide from TOC is being detected, gas flow in the digestion chamber is reversed, and the spent sample is carried out of the chamber to drain.

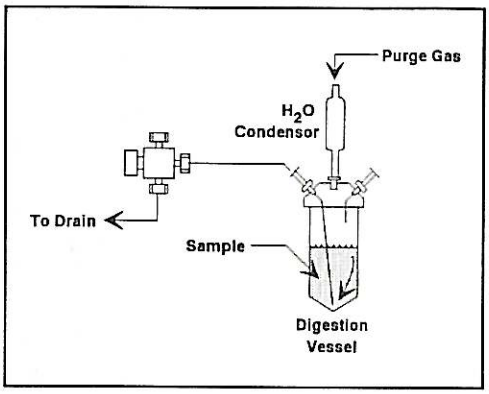


Fig. 4.9 Drain Step

Then purge gas flow is returned and a metered amount of acid is added to rinse the bottom of the digestion vessel. The system is then drained again and is ready for a new sample.

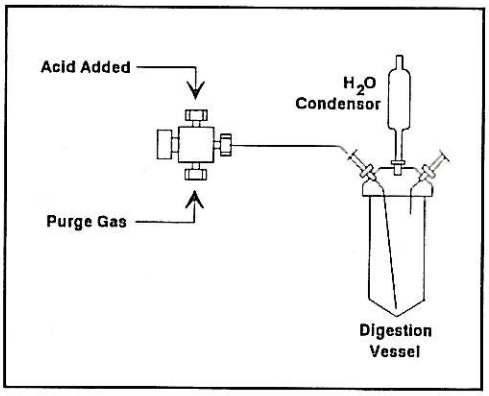


Fig. 4.10 Acid Rinse Step

Power Up

The Model 700 may be powered up in one of three states. These are:

- NORMAL POWER UP STATE
- #1 KEY POWER UP STATE
- #0 KEY POWER UP STATE

The **NORMAL POWER UP STATE** preserves previously set blank(s) and calibration data as well as volume, time, and alarm settings, but resets the temperatures, system configuration options, and sample numbers to their factory-set default values. The time of day value is reset to zero.

The **#1 KEY POWER UP STATE** preserves all the values which were in memory at the last power down except for the time of day. The time of day value is reset to zero.

The **#0 KEY POWER UP STATE** resets all the values in memory to their factory-set default values.

The choice of the power up state used depends on the analyses to be performed. The **NORMAL POWER UP STATE** allows the user to immediately begin running reagent blanks using normal time, temperature, and volume settings (sample pump and sample loop valve are disabled but auto-run is enabled).

The **#1 KEY POWER UP STATE** allows the user to immediately begin analyzing samples using the system configurations, volumes, times, and temperatures which were in use at the time of last power down. In this case, the user assumes that blank and calibration values will remain the same, a possibility which can be confirmed after extended operation of the instrument.

The **#0 KEY POWER UP STATE** allows the user to set every parameter back to default conditions to begin a completely new analysis scheme. The calibration factor is set to 1 so it must be re-entered or redetermined before analysis. The calibration constant is stable regardless of type of sample analysis, so redetermination of the calibration constant is generally not necessary for each analysis set. Because of this the **#0 KEY POWER UP STATE** is rarely used.

The Power Up States table lists the setting for each user-changeable parameter upon each of the three types of power up. See Fig. 4.11 on the next page.

NORMAL Power Up State

NOTE: Instrument will not power up without gas pressure of at least 15 psig.

- Confirm routing of drain line(s) to a waste receptacle.
- Turn on nitrogen gas flow and confirm 30 psi delivery pressure.
- If the Purgeable Organic Carbon Capability is installed, turn on the oxygen gas flow and confirm 10 psi delivery pressure.
- If a separate actuator gas is used, turn on the actuator gas flow and confirm 30 psi delivery pressure.
- Confirm that the reagent bottles are being purged and have sufficient reagents to perform the planned analyses.
- Turn on the power using the main POWER switch. Values in memory will be set to those listed in Fig. 4.11 under NORMAL POWER UP.

The following message will appear on the display screen:

```
TIC/TOC ANALYSIS
00:00:00  STANDBY  <
```

- Confirm that the purge and carrier nitrogen gas flow meters show the flow settings specified in **Chapter 1.**
- The screen will display the STANDBY message until the digestion vessel is heated to the proper temperature. When the digestion vessel reaches its preset

POWER UP STATES

Parameter	Default Value	NORMAL-	#1 KEY-	#0 KEY-
Analysis Mode	**TIC/TOC**	**Default**	**Last Val**	**Default**
TIC Blank	0	Last Val	Last Val	Default
TOC Blank	0	Last Val	Last Val	Default
POC Blank	0	Last Val	Last Val	Default
Standard Mass	0 ug	Last Val	Last Val	Default
Standard Average	1 mV	Last Val	Last Val	Default
Scaling Factor	1 ug C/mV	Last Val	Last Val	Default
Sample Volume	1 ml	Last Val	Last Val	Default
Acid Volume	200 ml	Last Val	Last Val	Default
Oxid Volume	1000 ml	Last Val	Last Val	Default
Auto Repeat Time	0:00:00	Last Val	Last Val	Default
Extended Reaction	0:00:00	Last Val	Last Val	Default
Extended Purging	0:00:00	Last Val	Last Val	Default
Time of Day	0:00:00	Default	Default	Default
Sample Pump Time	7 sec	Last Val	Last Val	Default
Prim Trap Temp	200°C	Default	Last Val	Default
POC Trap Temp	180°C	Default	Last Val	Default
Dig Blk Temp	100°C	Default	Last Val	Default
Furnace Temp	800°C	Default	Last Val	Default
Acid Injection	Enabled	Default	Last Val	Default
Oxid Injection	Enabled	Default	Last Val	Default
Sample Pump	Disabled	Default	Last Val	Default
Sample Loop Valve	Disabled	Default	Last Val	Default
Auto-Run	Enabled	Default	Last Val	Default
Auto-Print	Enabled	Default	Last Val	Default
Auto-Sample	Disabled	Default	Last Val	Default
Ready/Standby	Disabled	Default	Last Val	Default
Sample ID Number	01	Default	Last Val	Default
Spl Stop Number	00	Default	Last Val	Default
TIC Alarm Hi	0 ppm C	Last Val	Last Val	Default
TIC Alarm Lo	0 ppm C	Last Val	Last Val	Default
TOC Alarm Hi	0 ppm C	Last Val	Last Val	Default
TOC Alarm Lo	0 ppm C	Last Val	Last Val	Default
TIC Only	Disabled	Default	Last Val	Default
TC Only	Disabled	Default	Last Val	Default
Ampule Analysis	Disabled	Default	Last Val	Default
Wafertoc Analysis	Disabled	Default	Last Val	Default
POC Only	Disabled	Default	Last Val	Default

Fig. 4.11 Table of Power Up States

temperature, the following message will appear on the display screen:

This heating period is less than 10 minutes. If the RUN/STOP key is pressed during this period, the < in the STANDBY message will change to > and the instrument will wait until the preset digestion vessel temperature has been reached; then the analysis sequence will begin automatically.

```
TIC/TOC ANALYSIS
00:00:00 *READY* <
```

IMPORTANT: Press CLEAR key to assure that reagents will be carried to drain as they are dispensed.

- Open the keypad drawer and press the **PRIME ACID** key 10 to 20 times to prime the acid pump.
- Press the **PRIME OXIDANT** key 10 to 20 times to prime the oxidant pump.

#1 KEY Power Up State

- Confirm routing of drain line(s) to a waste receptacle.
- Turn on nitrogen gas flow and confirm 30 psi delivery pressure.

Instrument will not power up without gas pressure of at least 15 psig.

- If the Purgeable Organic Carbon Capability is installed, turn on the oxygen gas flow and confirm 10 psi delivery pressure.
- If a separate actuator gas is used, turn on the actuator gas flow and confirm 30 psi delivery pressure.
- Confirm that the reagent bottles are being purged and have sufficient reagents to perform the planned analyses.
- While pressing the **#1** key, turn on the power using the main POWER switch. Be sure to hold the **#1** key down until the display message appears. Values in memory will be those in memory upon the last power down as shown in Fig. 4.11 under #1 KEY POWER UP.

The following message will appear on the display screen:

Confirm that the purge and carrier nitrogen gas flow meters show the flow settings specified in **Chapter 1.**

```
TIC/TOC ANALYSIS
00:00:00  STANDBY  <
```

The screen will display the STANDBY message until the digestion vessel is heated to the proper temperature. When the digestion vessel reaches its preset temperature, the following message will appear on the display screen:

This heating period is less than 10 minutes. If the **RUN/STOP** key is pressed during this period, the < in the STANDBY message will change to > and the instrument will wait until the preset digestion vessel temperature has been reached; then the analysis sequence will begin automatically.

```
TIC/TOC ANALYSIS
00:00:00 *READY* <
```

IMPORTANT: Press CLEAR key to assure that reagents will be carried to drain as they are dispensed.

- Press the **PRIME ACID** key 10 to 20 times to prime the acid pump.
- Press the **PRIME OXIDANT** key 10 to 20 times to prime the oxidant pump.

#0 KEY Power Up State

Instrument will not power up without gas pressure of at least 15 psi.

- Confirm routing of drain line(s) to a waste receptacle.
- Turn on nitrogen gas flow and confirm 30 psi delivery pressure.
- If the Purgeable Organic Carbon Capability is installed, turn on the oxygen gas flow and confirm 10 psi delivery pressure.
- If a separate actuator gas is used, turn on the actuator gas flow and confirm 30 psi delivery pressure.
- Confirm that the reagent bottles are being purged and have sufficient reagents to perform the planned analyses.
- While pressing the **#0** key, turn on the power using the main POWER switch. Be sure to hold the **#0** key down until the display message appears. Values in memory will be set to default as shown in Fig. 4.11 under #0 KEY POWER UP.

The following message will appear on the display screen for 2 seconds:

```
MEMORY LOST USING
DEFAULT SETTINGS
```

followed by:

```
TIC/TOC ANALYSIS
00:00:00  STANDBY  <
```

- Confirm that the purge and carrier nitrogen gas flow meters show the flow settings specified in **Chapter 1.**
- The screen will display the STANDBY message until the digestion vessel is heated to the proper temperature. When the digestion vessel reaches its preset temperature, the following message will appear on the display screen:

This heating period is less than 10 minutes. If the **RUN/ STOP** key is pressed during this period, the < in the STANDBY message will change to > and the instrument will wait until the preset digestion vessel temperature has been reached; then the analysis sequence will begin automatically.

```
TIC/TOC ANALYSIS
00:00:00 *READY*  <
```

IMPORTANT: Press CLEAR key to assure that reagents will be carried to drain as they are dispensed.

- Open the keypad drawer and press the **PRIME ACID** key 10 to 20 times to prime the acid pump.
- Press the **PRIME OXIDANT** key 10 to 20 times to prime the oxidant pump.

Sample Introduction

Syringe Injection

The IR Detector is linearized over a range of 0 to 50 ug C. A syringe injection volume appropriate to introduce this range of carbon per sample should be selected, depending on the sample's carbon concentration. Use the table in Fig. 4.12 to select the proper sample injection volume.

The following steps describe the procedure for sample introduction by syringe injection.

Sample Carbon Concentration	Syringe Injection Sample Volume
0 ppb C - 5 ppm C	10 ml
20 ppb C - 10 ppm C	5 ml
50 ppb C - 25 ppm C	2 ml
100 ppb C - 50 ppm C	1 ml
200 ppb C - 100 ppm C	0.5 ml
500 ppb C - 250 ppm C	0.2 ml
1 ppm C - 500 ppm C	0.1 ml
2 ppm C - 1000 ppm C	50 ul
5 ppm C - 2500 ppm C	20 ul
10 ppm C - 5000 ppm C	10 ul
20 ppm C - 10000 ppm C	5 ul

Fig. 4.12 Sample Injection Volume Table

- Select the desired analysis mode and confirm that the SAMPLE PUMP, SAMPLE LOOP and AUTO-RUN options are DISABLED by pressing the **SET SYSTEM CONFIGURATION** key and reviewing the screen displays. These settings will remain until power is turned off. Settings of other options should remain at their default values. Upon power-up, these options may be reset to default conditions or to their last value.

- Confirm that appropriate calibration values are in memory by pressing the **SELECT DISPLAY MODE** key until CALIBRATION CONSTANTS is selected. Then press the **SELECT NEXT DISPLAY** key to successively review blank values and calibration constants. Calibration constants will remain in memory for at least 10 years when power is turned off.

- Press the **CLEAR** key to display the READY condition.

- Confirm that the sample drain line is run to an appropriate waste receptacle.

- Confirm that the sample volume is set to the volume injected by pressing the **SET SAMPLE VOLUME** key. Enter the correct number of milliliters injected if necessary and press the **ENTER** key.

- Press the **RUN/STOP** key to begin the analysis sequence.

- At 27 seconds, the INTRODUCE SAMPLE message appears on the screen, inject the sample into the sample injection port using a syringe with a 2" needle (22 gauge or thinner). Press the **RUN/STOP** key immediately after injection.

The CLEAR key may be pressed at any time to reset the timer, abort the present analysis, drain the sample, and bake the trap.

For greater precision, it is recommended that the sample be introduced into the injection port block, pressing the **RUN/STOP** key and allowing acid, as it is introduced, to rinse over the needle before it is removed from the injection port. This will help assure that all the sample is carried into the digestion vessel before the syringe is withdrawn.

- Near the end of the analysis sequence, the millivolt response to sample and the concentration in ppm C will alternately be displayed. The sequence will end with the display of the concentration.

- When the concentration is displayed and the lamp on the **RUN/STOP** key quits flashing, the analysis sequence is complete.

Loop Injection - Bottled Samples

The IR Detector is linearized over a range of 0 to 50 ug C. A sample loop of appropriate volume to introduce this range of carbon per sample should be selected. Use the table in Fig. 4.13 to select the correct loop according to sample carbon concentration.

Sample Carbon Concentration	Approximate Loop Volume
10 ppb C - 5 ppm C	10 cc
20 ppb C - 10 ppm C	5 cc
0.1 ppm C - 50 ppm C	1 cc
0.3 ppm C - 150 ppm C	0.34 cc
1 ppm C - 500 ppm C	0.10 cc (optional)
2 ppm C - 1000 ppm C	50 ul (optional)
3 ppm C - 1500 ppm C	35 ul (optional)

Fig. 4.13 Approximate Loop Volume Table

- Samples with carbon concentrations up to 150 ppm C can be analyzed with the standard process option valve (OI Part #164-559). Samples with concentrations greater than 150 ppm C should either be diluted or injected by syringe as described earlier in this chapter. The microliter loop option (OI Part #172-784) will allow analysis of up to 2500 ppm C with excellent precision. With either of the two process sampling valves, precision of better than ± 2% can be expected.

- Sample loops are installed on the standard process sampling valve by simply unscrewing the sample loop connectors (see Fig. 4.14) and installing the new loop. The connections are remade finger-tight.

- Microliter loops (i.e. 20 ul-340 ul), used with the microliter loop option, are designed to screw directly into the cap of the sampling valve at Ports 3 and 6. The nominal 340 ul sample loop (labeled 0.34 ml at the factory) is made from two "legs" which are equal lengths of 1/8" OD tubing and have plastic fittings on each end. When sample volumes of 0.34, 1, 5 or 10 milliliters are to be used, screw one "leg" of the 0.34 ml loop into Ports 3 and 6 of the sample valve cap respectfully and connect the two "legs" together (for 0.34 ml) or connect the 1, 5, or 10 milliliter loop to the legs with the plastic couplings provided for the desired sample range as described above. The 0.34 ml loop is a common volume already included in the 1, 5 and 10 milliliter loops when calibrated at the factory. Thus, it must be installed to prevent sampling errors that would cause lower than actual carbon concentration to be calculated.

Starting Loop Injections

The following steps describe the procedure for sample introduction using a sample loop.

- Confirm that the SAMPLE PUMP, SAMPLE LOOP and AUTO-RUN options are enabled by pressing the **SET SYSTEM CONFIGURATION** key and reviewing the screen displays. These settings will remain until power is turned off. Settings of other options should remain at their default values. Upon power-up, these options may reset to default conditions or to their last value.

- Confirm that appropriate calibration values are in memory by pressing the **SELECT DISPLAY MODE** key until CALIBRATION CONSTANTS is selected. Then press the SELECT NEXT DISPLAY key to successively review

blank values and calibration constants. Calibration constants will remain in memory for at least 10 years when power is turned off.

- Confirm that the appropriate sample loop is installed then set the loop volume in processor memory by pressing the SET SAMPLE VOLUME key and entering the value marked on the loop.

The volume of the sample loop valve "legs" (ca. 0.34 ml) is already included in the volume of the nominal 1 ml, 5 ml and 10 ml sample loops shipped from the factory for either the standard or microliter loop valves. Thus, the volume entered from the keypad is the volume marked on the sample loop.

- Press the **ENTER** key to display READY message.

- Connect the teflon sampling tube to the Luer-Lok fitting in the recessed front panel near the injection port. Place the free end of the tube in the sample.

- Confirm that the loop drain and sample drain lines are run to an appropriate waste receptacle.

- Press the **RUN/STOP** key to begin analysis. The sample will be pulled into the loop and then injected into the reaction vessel.

- After the beginning of the time sequence, leave the sampling tube in the sample bottle for automatic repetitive analysis, or transfer to a new bottle for the next sample. The sample pump can be manually activated between samples to eliminate the possibility of cross contamination by water in the end of the sample tube which can diffuse into the new sample bottle. In this case, air, rinse water, or the next sample can be manually pumped through the sampling tube.

- Near the end of the analysis, the millivolt response of sample and the measured carbon concentration will be alternately displayed. The concentration display should be manually recorded unless the printer is connected. With the auto-run option enabled, the timer will reset and the next sample will be injected to begin the next analysis, and the screen display of carbon concentration will be erased. The screen display can be recalled using the . (decimal) key.

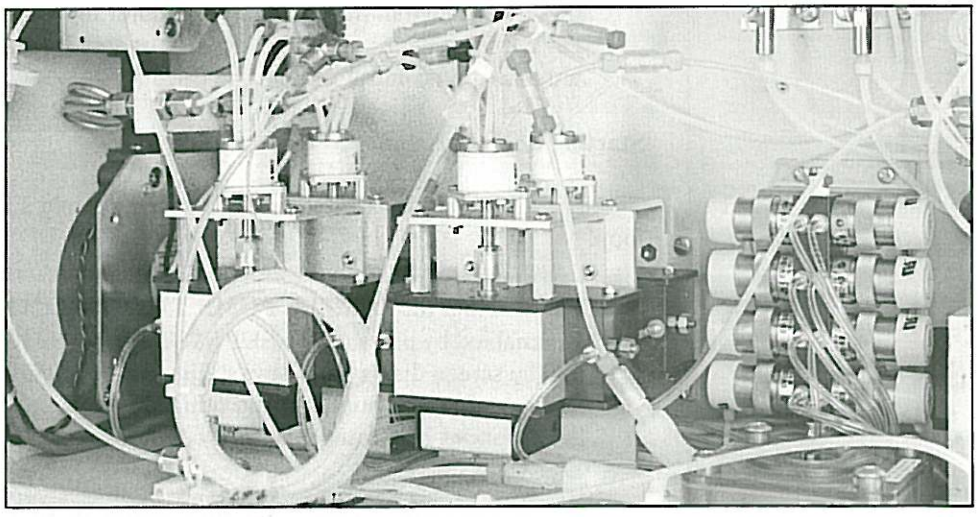


Fig. 4.14 Sample Loop Connections

Ending Loop Injections

To discontinue auto-run analysis, perform any one of the following steps:

- Enter a sample stop number corresponding to the last sample number to be analyzed.
- After the final sample has been introduced into the digestion vessel (at analysis time 1 min 10 sec), disable the auto-run option.
- Pressing the **CLEAR** key at anytime will reset the analysis timer to zero, abort the current analysis sequence, drain the sample, bake the trap(s) and clear Last Analysis values (only) from display memory.

Loop Injection - On-Line Sampling

Water may be sampled from flowing processes if the sample loop is plumbed on-line with the process water. If the process water pressure is sufficient (5 psi or greater) the water pressure can be used to flow sample continually through the sample loop. In this case, the sample pump down-stream of the loop must be removed from the flow-line by by-passing the pump completely. This is accomplished by connecting the finger-tight fittings, which are normally connected to the pump inlet and outlet, together and disabling the sample pump function.

Note: Maximum water feed pressure for process sampling is 60 psi.

This pump is included in the Process Sampling Capability (OI Part #164-559). It is used to aspirate sample through the loop sampling inlet and the sample loop. The pump should be by-passed during pressure-fed flow-through (on-line) sampling to avoid creating back pressure. If, however, the process water pressure is not sufficient (less than 5 psi) to force sufficient flow of water through the sample loop, the pump may be used to pull water from the process stream to fill the loop. This is accomplished by first disabling the sample pump function and then manually turning the pump on by pressing the **SAMPLE PUMP ON/OFF** key, thus allowing the sample pump to run continuously.

In either case, the process water flow line is connected to the loop sampling Inlet Port using a Luer-Lok fitting. Then follow the instructions outlined for "Loop Injection-Bottled Samples" except that the sample pump function will be disabled.

Loop Injection - With Autosampler

Sample loop injection with the autosampler is identical to the procedure described for "Loop Injection-Bottled Samples" after the autosampler has been installed. The autosampler is programmed to automatically rinse the sample loop and associated tubing between samples using the wash station.

To analyze samples using the autosampler, enable the autosampler using the **SET SYSTEM CONFIGURATION** key and follow the instructions outlined in the section Loop Injection - Bottled Samples.

Loop Injection - Air or Gas Sampling

Samples of air or gas can be analyzed in a manner identical to water samples according to any of the previous sections except that gas is introduced rather than water and the acid and oxidant pumps are disabled using the **SET SYSTEM CONFIGURATION** key.

Running Reagent Blanks

Concept

If a complete analysis sequence is performed (i.e. react carbon/purge CO_2/detect CO_2) without injection of a sample, the detector responses for TIC and TOC (and POC when in operation) will still be generated due to carbon in the reagents, gas, tubing, and digestion chamber. These reagent blanks can be reduced to a minimum, consistent values, but cannot be completely eliminated. When standards or samples are analyzed, we make the assumption that the detector response generated from that analysis includes response due to this reagent blank in addition to response due to carbon in the sample. If the reagent blanks for TIC, TOC, and POC are determined prior to the analysis of standards or samples, the blank values may be subtracted from the detector response due to samples, to accurately determine the amount of carbon due only to the sample. Thus, reagent blanks should be run until replicate values are consistent, prior to sample analysis, using the same conditions of analysis as planned for the samples. Conditions of analysis are generally constant for routine samples but time, temperature, and volume parameters may be varied.

Procedure

- Power up.
- The instrument will be in the TIC/TOC Analysis Mode upon power up. If desired, press the **SELECT ANALYSIS MODE** key to select POC/TIC/TOC Analysis Mode.
- If default settings for any volumes, times, and temperatures are not desired, set any new conditions of analysis.
- Press the **SET SYSTEM CONFIGURATION** key. Press the **ENTER** key to advance to the next option. Confirm that the SAMPLE PUMP and SAMPLE LOOP are disabled, and that the AUTO-RUN, ACID PUMP, and OXIDANT PUMP are enabled.
- Press the **ENTER** key to advance through the groups and set other analysis options (i.e. TIC only, TC only, POC only, Ampule or Wafertoc) or press the **CLEAR ENTRY** key to return to the analysis display.
- Press the **SELECT DISPLAY MODE** key twice to advance the Display Mode to Calibration Constants.
- Press the **SELECT NEXT DISPLAY** key repeatedly to advance the display to the IC Blank Value.
- If new blank values are to be determined, set the IC Blank to zero by pressing the **0** (zero) key then the **ENTER** key. Likewise reset the OC (and POC) blank.

If the analyst is going to run TC, ampules or the Wafertoc analysis, the millivolt value will be entered as the OC blank.

If blank values in memory are not set to zero, display of subsequent millivolt values will represent the DIFFERENCE of the analyzed blank and the blank in memory. For example, a blank analysis reading of 2 mV always means that the analyzed blank is 2 mV more than the value in memory. Blank values need not be set to zero before blank determination if the analyst understands to ADD (or subtract, if negative) the average displayed value to (or from) the value in memory after blanks are determined by replicate analyses.

- Press the **SELECT DISPLAY MODE** key twice more to advance the Display Mode back to Normal.

- Press the **RUN/STOP** key to start the blank run.

- At the end of the blank run, the detector response in millivolts will be displayed for both TIC and TOC blanks on the screen, and should be recorded by the analyst unless a printer is connected to record values. The . (decimal) key may be used to recall last values.

- Allow the instrument to run replicate blanks until millivolt values are consistent.

- The IC and POC blank millivolt values should be in the range of 1 to 2 millvolts for acid injection volumes between 0 and 500 ul. If values above this range are observed (refer to the troubleshooting guide in Chapter 6 describing high blanks). Blank values should not vary more than ± 0.2 millivolts.

- The OC blank millivolt values should be in the range of 4 to 10 millivolts for oxidant injection volumes between 0 and 1000 ul but will increase by 6 to 10 millivolts when higher volumes of oxidant are added. Blank values should not vary more than ± 0.5 millivolts. If a high or unstable OC blank is encountered, refer to the troubleshooting guide in Chapter 6 describing that problem. The OC blank does not increase linearally with the volume of oxidant. A significant percentage of the blank is apparently due to CO_2 desorbed from the walls of the hot teflon digestion vessel. Another fraction comes from the tubing and gas. As the system is operated over a period of several months, the fraction of the OC blank due to these two sources will likely decrease.

- When blanks are consistent, they should be averaged and entered into memory. Access to blank values is the same as described earlier in the Calibration Constants display mode. See Fig. 4.15 and Fig. 4.16 for typical blanks chart recorder trace and printout.

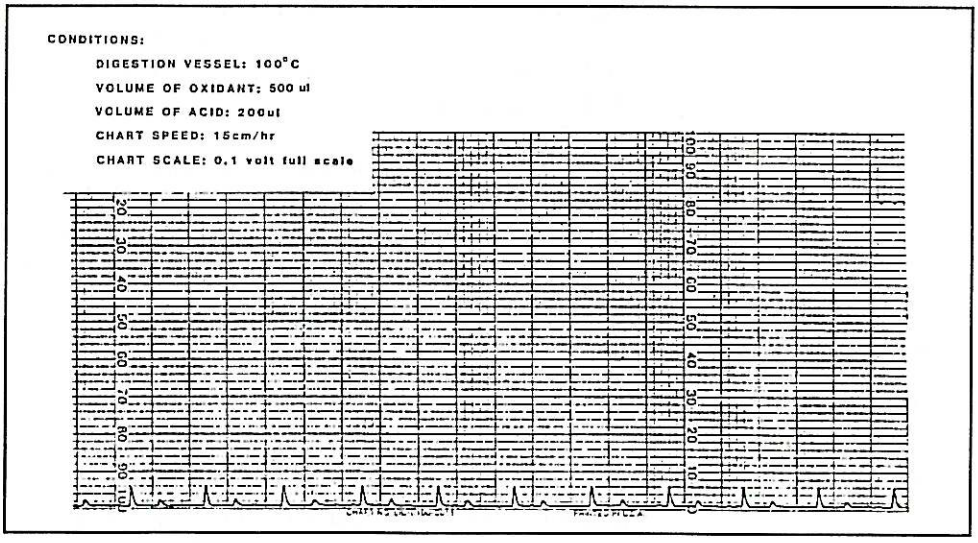


Fig. 4.15 Amplified Chart Trace of Blanks and Base Line

- After the instrument has been in operation for an extended period of time, blank values may be so consistent that redetermination of blank values continually yields near 0 mV when prior blank values are left in memory. This indicates

that the DIFFERENCE in the redetermined blank and the prior blank is negligible. In this case, the user may conclude that blank runs are not essential prior to analysis of each new sample set.

- Disable the auto-run option to stop blank determinations, or the **CLEAR** key may be pressed at any time to reset the timer, abort the present blank analysis, drain the reagents, and bake the trap.

SAMPLE ID	TIME OF DAY	IR RESPONSE	CALC. MASS C	CALC.CONC.
SPL# 00001	08:27:48	TIC = 1.70000 mV	0.08500 ug C	0.00850 ppm
SPL# 00001	08:32:48	TOC = 5.65000 mV	0.28250 ug C	0.03050 ppm
SPL# 00002	08:35:53	TIC = 1.50000 mV	0.07500 ug C	0.00750 ppm
SPL# 00002	08:48:53	TOC = 6.10000 mV	0.30550 ug C	0.02825 ppm
SPL# 00003	08:43:58	TIC = 1.50000 mV	0.07500 ug C	0.00750 ppm
SPL# 00003	08:48:58	TOC = 5.65000 mV	0.28250 ug C	0.02825 ppm
SPL# 00004	08:52:03	TIC = 1.65000 mV	0.08250 ug C	0.00825 ppm
SPL# 00004	08:57:03	TOC = 5.40000 mV	0.27000 ug C	0.02700 ppm
SPL# 00005	09:00:08	TIC = 1.55000 mV	0.07750 ug C	0.00775 ppm
SPL# 00005	09:05:08	TOC = 5.10000 mV	0.25500 ug C	0.02550 ppm
SPL# 00006	09:08:13	TIC = 1.55000 mV	0.07750 ug C	0.00775 ppm
SPL# 00006	09:13:13	TOC = 5.05000 mV	0.25250 ug C	0.02525 ppm
SPL# 00007	09:16:18	TIC = 1.50000 mV	0.07500 ug C	0.00750 ppm
SPL# 00007	09:21:18	TOC = 4.85000 mV	0.24250 ug C	0.02425 ppm
SPL# 00008	09:24:23	TIC = 1.55000 mV	0.07750 ug C	0.00775 ppm
SPL# 00008	09:29:23	TOC = 4.85000 mV	0.24250 ug C	0.02425 ppm
SPL# 00009	09:32:28	TIC = 1.70000 mV	0.07500 ug C	0.00750 ppm
SPL# 00009	09:37:28	TOC = 5.65000 mV	0.26750 ug C	0.02675 ppm
SPL# 00010	09:40:33	TIC = 1.55000 mV	0.07750 ug C	0.00775 ppm
SPL# 00010	09:45:33	TOC = 5.30000 mV	0.26500 ug C	0.02650 ppm

Fig. 4.16 Typical Printout of Blank Replicates

Calibration

Concept

The instrument measures peak heights of signals generated by the IR from blanks, standards, and samples. Reagent blanks are determined first, by repetitive cycling of the analysis sequence without introduction of standard or sample. Millivolt signals from these blank runs are recorded and averaged. Average millivolt values of IC, POC, and OC reagent blanks are entered into memory.

The millivolt blank value in memory is automatically subtracted from every millivolt measurement during analysis, before any other calculations, so that the millivolt readings displayed for blanks, standards, and samples have previously had IC, POC, and OC blanks subtracted.

Replicates of a calibration standard are run and millivolt values recorded. The mass of carbon introduced is entered into memory from the keyboard. The average millivolt value of the calibration replicates is entered into memory and a scaling factor (number of micrograms carbon per millivolt response) is calculated by the processor.

Calibration with a purgeable standard is not recommended due to difficulties in preparing and maintaining a constant concentration in aqueous solution.

The processor then multiplies each subsequently measured millivolt response by this scaling factor to calculate the number of micrograms carbon (mV x ug C/mV=ug C). The processor then divides this number of micrograms by the sample volume in memory to calculate the concentration (ug C/ml = ppm C). The millivolt response, micrograms carbon, and concentration for TIC and TOC are displayed and optionally printed. Because the IR detector is measuring carbon dioxide which has desorbed from a trap, peak shapes of TIC, TOC and POC are identical and the same calibration factor can be used for calculations involving any of these three carbon measurements. This single-point calibration and initial subtraction of blank millivolt response can be used because the IR detector response has been linearized over a range from 0 to 50 micrograms carbon as carbon dioxide. The single-point calibration is valid for any measurement which falls in this range. Samples yielding higher ug C values should be re-run using less sample or a multi-point calibration should be applied.

TIC Method

Using Sodium Carbonate

For calibration using inorganic carbon (TIC), a known volume of a solution of sodium carbonate is injected. One recommended calibration solution is 1000 ppm C in distilled or deionized water. Each microliter of this solution contains 1.0 microgram of carbon so that the injection of a specific number of micrograms of carbon is accomplished by injection of that many microliters of solution. The number of micrograms of carbon injected should be similar to the mass of carbon expected for samples. For low-level analyses, a calibration solution of 100 ppm C may be more suitable so that larger volumes may be injected for better precision. In either case, correction for the carbon in the reagent water is unnecessary because it is less than 0.1% of the total carbon in the solution. Solutions of sodium carbonate are basic and will absorb CO_2 from the atmosphere, so they should remain sealed when not being sampled, and unused standard solutions should be disposed of properly after no more than 3 weeks.

Using Pure Carbon Dioxide Gas

The instrument may be calibrated by injecting pure CO_2 with a gas-tight syringe. At normal temperature and pressure, each microliter of CO_2 contains almost exactly 0.5 micrograms carbon, as seen from the following calculation:

$$\frac{12\text{g C} \quad \text{x} \quad 1\text{ mole } CO_2 \text{ x } 273 \text{ x } P}{1\text{ mole } CO_2 \quad \text{x} \quad 22.4\text{ l } CO_2 \text{ x } T \text{ x } 760} = 0.5\text{ g C/l } CO_2$$

Where T and P are normal temperature (°K) and pressure (mmHg).

Using Bottled Air

The instrument may be calibrated using the CO_2 in a bottle of compressed air provided that the CO_2 content of the air is determined using a primary standard (sodium carbonate or KHP solution).

TOC Method

Calibration using organic carbon is accomplished in a manner similar to the TIC calibration except an organic compound such as potassium hydrogen phthalate (KHP) is used. Standard solutions of 1000 ppm C or 100 ppm C in distilled or deionized water are recommended to eliminate the need for correction of organic carbon in the water (distilled water generally has less than 200 ppb C). TOC standards can be compared to TIC standards as a check of oxidation efficiency. The organic carbon calibration sequence takes eight minutes.

Constant Volume Method

The routine calibration methods typically call for injection of 5 to 50 microliters of standard solution, whereas sample volumes of 0.5 to 10 ml are used for all but high TOC (> 100 ppm C) samples. If calibration using the same volume of standard as used for samples is desired, a standard solution of carbon concentration similar to the samples may be prepared (using TIC or TOC). The number of micrograms carbon entered from the keyboard during the calibration procedure is calculated by multiplying the concentration of standard in ppm C (disregarding the contribution from the reagent water) by the volume in milliliters of the standard injected (example: 5 ppm C standard x 5 ml sample loop = 25 ug C). If a sample loop is used, the volume written on the loop is the multiplier. If a microliter syringe is used, multiply the number of

microliters by 0.001. If the carbon concentration of the dilution water is significant with respect to that of the prepared standard, the dilution water can be first analyzed and millivolts averaged, then the standard. The difference in the two millivolt averages can then be entered for calibration (see Standard Additions later in this chapter as an example for this technique). Calibration in this manner allows unattended replicates of the standard to be run if the sample loop option is used. Either the TIC or TOC calibration mode can be used in this manner, depending on the nature of the standard material.

Calibration Procedures

CALIBRATE MODE (Analyst Assisted/Syringe)

The CALIBRATE MODE is an analyst assisted calibration routine where through use of displayed screen prompts the analyst is systematically stepped through the calibration sequence.

- Power up and determine reagent blanks.
- Press the **SET SYSTEM CONFIGURATION** key. Press the **ENTER** key to advance to next option. Confirm that ACID PUMP and OXIDANT PUMP are enabled and SAMPLE PUMP, SAMPLE LOOP and AUTO-RUN functions are disabled.
- Press the **SELECT ANALYSIS MODE** key to select CALIBRATE MODE.
- Choose key **1** if a TIC standard (i.e. Sodium Carbonate) is to be used or key **2** if a TOC standard (i.e. KHP) is to be used.

It does not matter what method of calibration you choose in terms of the analysis mode that will be used to run your samples. The calibration constant obtained by either the TIC method or TOC method can be used to run TIC, POC, TOC, TC, Ampule or Wafertoc analysis. The only difference is that the TIC method takes 4 minutes per standard run and the TOC method takes 8 minutes per standard run.

If these values were entered as described previously, they need not be re-entered here.

- Enter the average millivolt value for the IC blank obtained, then likewise for the POC blank (if POC is to be run on samples) and the OC blank.
- When the OC blank is entered the display will change to:

ENTER STD MASS 00000 ug C

Numerical entries are always followed by pressing the ENTER key. If no numerical entry is made, the system will use the number displayed when the ENTER key is pressed, and the next instruction will appear.

- Enter the amount of micrograms carbon to be injected as the standard and press the **ENTER** key.

The micrograms of carbon is determined by:

concentration of standard (ppm C) x volume injected (ml) = STD MASS (ug C)

(where ug C is less than or equal to 50)

- Press the **RUN/STOP** key to begin analysis and inject the standard when prompted by the screen.

- At the end of the calibration run, the detector millivolt response for the standard will be displayed on the screen and should be recorded by the analyst unless a printer is connected to record the values. The . (decimal) key can be used to recall last values.

- Run replicate standards as prompted by the screen until satisfactory duplication of results are obtained.

- Press the **SELECT NEXT DISPLAY** key to display:

ENTER STD AVG 00000 mV

- Calculate the average millivolt response for the standard and enter the value from the keypad, then press the **ENTER** key.

- When the **ENTER** key is pressed after keying in the average millivolt response, the scaling factor (ug C/mV) will be automatically calculated and displayed. Press the **ENTER** key to store this constant in processor memory. Display will show:

CALIBRATION COMPLETE SELECT ANALYSIS MODE

- Choose TIC/TOC or POC/IC/TOC mode depending on analysis to be performed on samples.

Calibration Procedure - TIC/TOC or POC/IC/TOC Mode (Unattended/Loop Sampling)

The procedure described in this section provides for the most efficient means of calibration. Except for numerical entries of constants, this procedure is automatic. It is also desirable since the calibration routine used is a facsimile for the analysis of the samples to be run after calibration is complete. If your instrument does not have the Process Sampling Option (OI Part #164-559) the calibration sequence described here can still be used by disregarding notations concerning the sample loop, sample pump and auto-run functions which will always be disabled for instruments not having the Process Sampling Option.

- Power up and set all volumes to be used for analysis, then run reagent blanks.

Press 1 to enable a function that is disabled.

- Press the **SET SYSTEM CONFIGURATION** key. Press the **ENTER** key to advance to the next option. Confirm that the ACID PUMP, OXIDANT PUMP, SAMPLE PUMP and SAMPLE LOOP VALVE functions are enabled by successively pressing the **ENTER** key.

TIC only, TC only, AMPULE ANALYSIS and WAFERTOC ONLY can be run in either of the analysis modes. POC only can be performed only in the POC/IC/TOC analysis mode.

- Continue to press the **ENTER** key until the AUTO-RUN option appears on the screen and confirm that it is enabled.

- Press the **SELECT ANALYSIS MODE** key to choose the desired mode of operation (i.e. TIC/TOC or POC/IC/TOC).

- Press the **SELECT DISPLAY MODE** key to advance the Display Mode to CALIBRATION CONSTANTS.

If these values were entered as described earlier, they need not be re-entered here.

- Press the **SELECT NEXT DISPLAY** key to advance the display to:

ENTER IC BLANK 00000 mV

- Enter the average millivolt value for the IC blank, then likewise for the POC blank (if POC is to be run on samples) and the OC blank.

- Press the **SELECT NEXT DISPLAY** key to advance the display to:

ENTER STD MASS 00000 ug C

- Enter the amount of micrograms carbon to be injected as the standard and press the **ENTER** key.

The microgram of carbon is determined by:

$$\frac{\text{Concentration of Standard (ppm C)} \times \text{Volume Injected (ml)}}{} = \text{STD MASS (ug C)}$$

(where ug C is less than or equal to 50)

- Press the **SELECT DISPLAY MODE** key to advance the DISPLAY MODE to NORMAL.

- Place sipper tube from the process sampling port into the container with the standard to be analyzed.

- Press the **RUN/STOP** key to begin analysis. The analysis of the calibration standard from this point is automatic and the analyst need not be present until several replicates of the standard have been analyzed.

- If a printer is not being used, the millivolt values will need to be recorded at the end of each run. These values may also be obtained by pressing the decimal key at the end of the run.

- When several satisfactory duplications of responses are obtained (typically 3), press the **SET SYSTEM CONFIGURATION** key, then advance to the AUTO-RUN option by pressing the the **ENTER** key and disable the function.

- Press the **SELECT DISPLAY MODE** key to advance to CALIBRATION CONSTANTS.

- Press the **SELECT NEXT DISPLAY** key to advance the display to:

ENTER STD AVG 00000 mV

- Calculate the average millivolt response from the replicate analysis and enter the value as the STD AVG. When the **ENTER** key is pressed, the scaling factor (ug C/mV) will automatically be calculated and displayed. Press the **ENTER** key to store this constant in processor memory. Press the **SELECT NEXT DISPLAY** key.

This completes the calibration sequence and samples may now be run as described in the following sections.

Running Water Samples for TIC and TOC

A summary of the general TIC/TOC method was given in the previous section. Methods listed here each involve some portion of that general description.

TIC/TOC

The TIC/TOC Analysis Mode includes the introduction of sample, addition of acid, purging and trapping of TIC, detection and display of TIC, addition of oxidant, oxidation of organics, purging and trapping of CO_2 from organic carbon, detection and display of TOC, draining of spent sample, and acid rinse of reaction vessel. This sequence of steps is accomplished in 8 minutes. Follow these steps to operate in the TIC/TOC mode:

- The instrument powers up in the TIC/TOC Analysis Mode. If the instrument is in another Analysis Mode, press the **SELECT ANALYSIS MODE** key to select TIC/TOC Analysis Mode.
- Confirm that TIC-Only and TC-Only options are disabled (Group 3 of SET SYSTEM CONFIGURATION).
- Follow instructions in sections Sample Introduction and Running Reagent Blanks for analysis of samples.

TIC-Only

The TIC-Only mode of analysis includes introduction of sample, addition of acid, purging and trapping of TIC, detection and display of TIC, draining of spent sample, and acid rinse of reaction vessel. Oxidant is not added. This sequence of steps is accomplished in three minutes. Follow these steps to operate in the TIC-Only mode:

- The instrument powers up in the TIC/TOC Analysis Mode. If the instrument is in another Analysis Mode, press the **SELECT ANALYSIS MODE** key to select TIC/TOC Analysis Mode.
- Enable TIC-only using the **SET SYSTEM CONFIGURATION** key (TIC-Only is in the Group 3 options).
- Follow instructions in sections Sample Introduction and Running Reagent Blanks for analysis of samples.

TC-Only

The TC-Only mode of analysis includes introduction of sample, addition of acid and oxidant, oxidation of organics, purging and trapping CO_2 from inorganic and organic carbon, detection and display of TC, draining of spent sample, and acid rinse of reaction vessel. Inorganic carbon is not purged from the sample after introduction, but is combined with TOC. This sequence of steps is accomplished in six minutes and can be used as a rapid TOC analysis if the samples are acidified and purged to remove TIC prior to sample introduction. Follow these steps to operate in the TC-Only mode:

- The instrument powers up in the TIC/TOC Analysis Mode. If the instrument is

in another Analysis Mode, press the **SELECT ANALYSIS MODE** key to select TIC/TOC Analysis Mode.

- Enable TC-only using the **SET SYSTEM CONFIGURATION** key (TC-Only is in the Group 3 options).

- Follow instructions in sections Sample Introduction and Running Reagent Blanks for analysis of samples.

TOC-Only

The TOC-Only mode of analysis is performed by selecting the Calibrate Mode using the **SELECT ANALYSIS MODE** key. It is identical to the TIC/TOC mode except that the TIC is purged to vent rather than being trapped for detection which is advantageous when analyzing samples with very high TIC compared to the TOC. The sequence of steps is accomplished in seven minutes. Follow these steps to operate in the TOC-Only mode:

- Select Calibrate Mode by pressing the **SELECT ANALYSIS MODE** key.

- Select TOC Calibrate by following the screen prompt.

- Follow instructions in sections Sample Introduction and Running Reagent Blanks for analysis of samples.

Specialized Samples

Extended Purge Time

Extended purge will affect Ampule Purge time when that option is selected.

The time duration of purging TIC in a routine carbon analysis has been optimized (2.5 mins.) to yield accurate results in the fastest time possible for a wide range of samples. Samples which contain TIC/TOC ratios of 1000 or more and caustic samples may require a longer purging time after acidification to completely remove TIC from the reaction vessel prior to TOC oxidation. If the POC option is installed and samples contain a high POC fraction and/or volatile species which are difficult to purge out of the sample, then extended purge time will be necessary for accurate recovery of the POC. Incomplete TIC and/or POC removal can result in erroneously high and non-reproducible TOC values. The purge time after acidification may be increased (typically 1-2 minutes but user-variable from 1 second to 24 hours) by pressing the **SET TIMES** key and advancing to the Extended Purge display using the **SELECT NEXT DISPLAY** key. This option may be reviewed and changed at any time during the analysis sequence. The extended purge time affects the purging of TIC and POC (if option is installed) and the associated IC and POC blanks. It does not affect the purging after TOC digestion.

Extended Reaction Time

Extended reaction will affect Wafertoc analysis reaction time when that option is being used.

The time duration of the chemical oxidation (3 mins.) of organic carbon in a routine analysis has been optimized to yield accurate results in the fastest time possible for a wide range of samples. Samples which contain more than 5% chloride ion (such as brines, concentrated HCl, and other chloride reagent solutions) may require a greater volume of oxidant and/or a longer oxidation period. This is because chloride ion competes with the organic carbon in the oxidation reaction. Incomplete oxidation of organic carbon results in erroneously low but fairly reproducible TOC values. The oxidation efficiency in a particular sample matrix may be checked by injection of a

known mass of organic carbon standard along with the sample. The reaction time may be increased (typically for 1-5 minutes but user-variable from 1 second to 24 hours) by pressing the **SET TIMES** key and advancing to the EXTENDED REACTION display using the **SELECT NEXT DISPLAY** key. The extended reaction time affects the reaction time of TOC and TC only analysis and the associated OC blank. This option may be reviewed and changed at any time during the analysis sequence.

Non-Linear and Overrange Results

Non-Linear (NL) Results

If the detector's millivolt response for an analysis exceeds 1000 mV, the display screen will give the message:

WARN: LINEARITY ERR
XXXXXX mV

and the letters "NL" will be printed (if printer is being used) beside the results. These values are in the non-linear range of the detector and resulting concentration values will be slightly low. In this case, a smaller sample size should be used or the sample diluted.

Over-Range (OR) Results

If the detector's millivolt response for an analysis exceeds 1600 mV the display screen will give the message:

WARN: IR SATURATED
XXXXX mV

and the letters "OR" will be printed (if printer is being used) beside the results. These values are in excess of that needed to saturate the detector and resulting concentration values will be significantly low. In this case, a smaller sample size should be used or the sample diluted.

Running Water Samples for POC

POC/TIC/TOC

A water sample may be introduced into the instrument either by syringe injection...

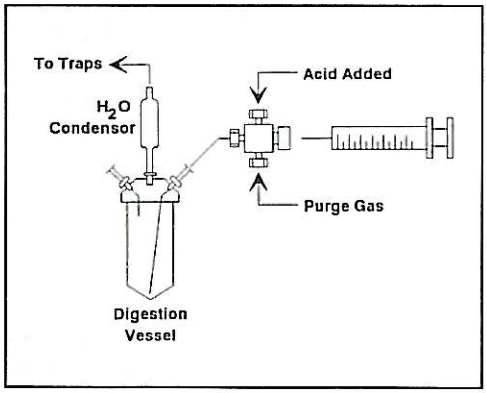


Fig. 4.17 Sample Introduction by Syringe

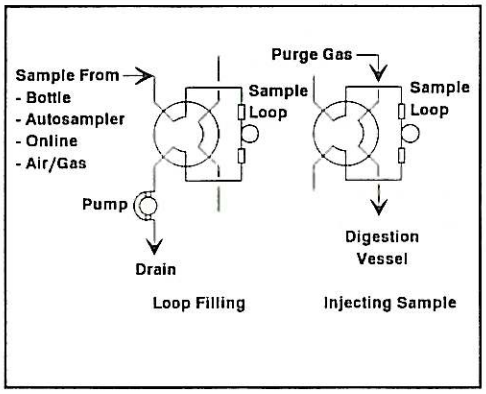


Fig. 4.18 Sample Introduction by Sample Loop

...or by means of a calibrated sample loop.

Sample introduction is followed by a metered amount of acid into the digestion vessel.

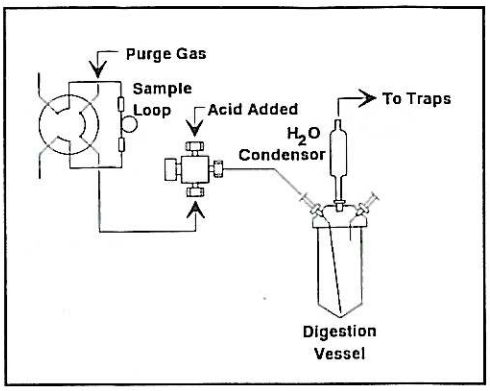


Fig. 4.19 Acid Addition Step

After acid has been added, a gas stream purges out any carbon dioxide formed from inorganic carbon and any purgeable organic carbon (POC) in the sample. POC is trapped on a Tenax GC trap held at 25°C where as CO_2 passes through. The carbon dioxide is carried to a molecular sieve trap held at 25°C where it is also trapped and concentrated.

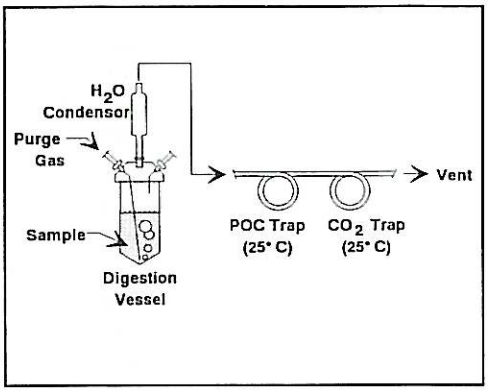


Fig. 4.20 Purge & Trap Step

When purging and trapping is complete, the CO_2 trap is placed in-line with the infrared detector and rapidly heated to 200°C. A stream of gas desorbs the carbon dioxide from the trap and carries it into the detector. The detector response represents the amount of TIC in the sample. Concentration of TIC is displayed and may be automatically printed.

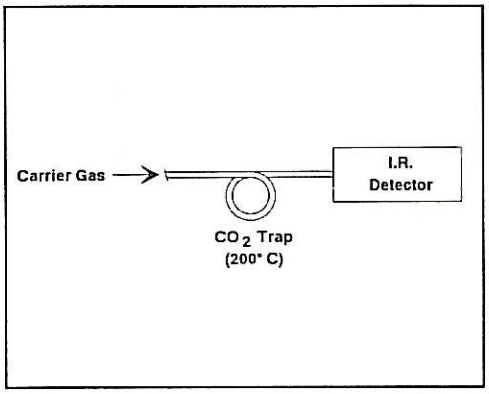


Fig. 4.21 Desorption/Detection Step

Then the POC trap is placed in-line with a furnace and the detector and rapidly heated to 180°C. The POC is oxidized under oxygen flow at 800°C in a catalysis tube to CO_2 and carried to the detector. The detector response represents the amount of POC in the sample. Concentration of POC is displayed and may be printed.

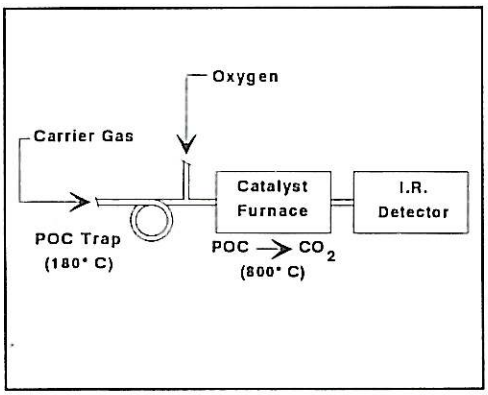


Fig. 4.22 POC Desorption/Detection Step

While the carbon dioxide from TIC and POC is being detected, a metered amount of sodium persulfate is added to the sample, which by now has been heated to 100°C. Purge gas flow to the digestion vessel has been shut off. The persulfate reacts with organic carbon in the sample to produce carbon dioxide, which accumulates in the digestion vessel.

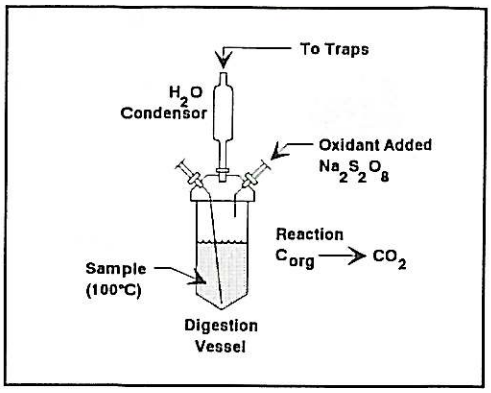


Fig. 4.23 Oxidant Step

After a preset reaction time, the digestion vessel is placed in-line with the trap and a gas stream purges out any carbon dioxide produced by the persulfate oxidation. This carbon dioxide is carried to the trap held at 25°C where it is trapped and concentrated.

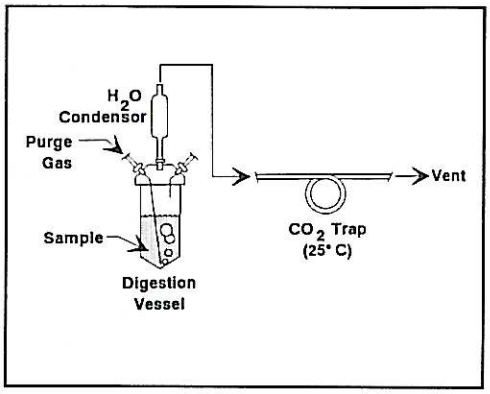


Fig. 4.24 Purge & Trap Step

As before, the trap is then placed in-line with the detector and heated. The carbon dioxide is carried into the detector, and the resulting concentration of TOC in the sample is displayed/printed.

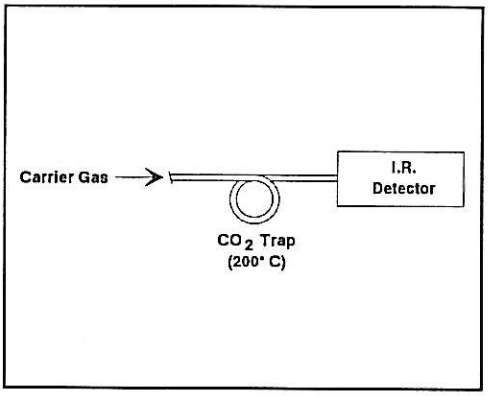


Fig. 4.25 Desorption/Detection Step

While the carbon dioxide from TOC is being detected, gas flow in the digestion chamber is reversed, and the spent sample is carried out of the chamber to drain.

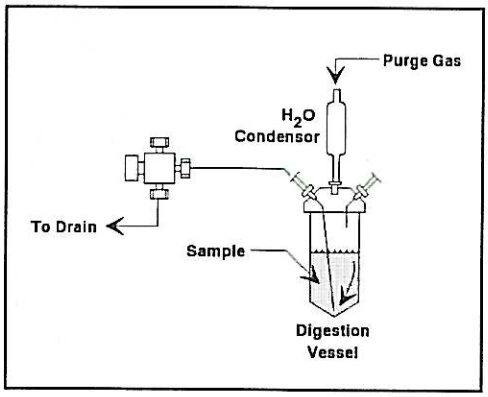


Fig. 4.26 Drain Step

Then purge gas flow is returned and a metered amount of acid is added to rinse the bottom of the digestion vessel. The system is then drained again and is ready for a new sample. The sequence of steps is accomplished in 10 minutes.

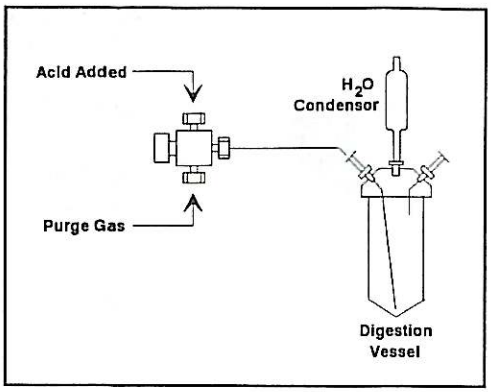


Fig. 4.27 Acid Rinse Step

Follow these steps to operate in the POC/TIC/TOC modes:

- Select POC/TIC/TOC Analysis by pressing the **SELECT ANALYSIS MODE** key.
- Press the **SET SYSTEM CONFIGURATION** key and confirm that all Group 3 options are disabled.
- Follow instructions in sections Sample Introduction and Running Reagent Blanks for analysis of samples.

POC-Only

The POC-Only mode of analysis includes introduction of sample, addition of acid, purging and trapping of POC, detection and display of POC, draining of spent sample, and acid rinse of reaction vessel. Oxidant is not added. This sequence of steps is accomplished in five minutes. Follow these steps to operate in the POC-Only mode:

- Select POC/TIC/TOC Analysis by pressing the **SELECT ANALYSIS MODE** key.
- Enable the POC-Only Analysis by pressing the **SET SYSTEM CONFIGURATION** key (POC-Only is a Group 3 option).
- Follow instructions in sections Sample Introduction and Running Reagent Blanks for analysis of samples.

Ampule Analysis

Concept

Ampule Analysis includes preparation of samples inside glass ampules, digestion of the ampules to oxidize TOC, purging and trapping of CO_2 from TOC, and detection and display of TOC. The preparation and digestion of ampules for TOC is described in a separate Ampule Purging and Sealing Unit manual. Ampules prepared according to this procedure are connected to an Ampule Breaking Assembly on the front of the Model 700 and processed according to the Ampule Analysis Mode.

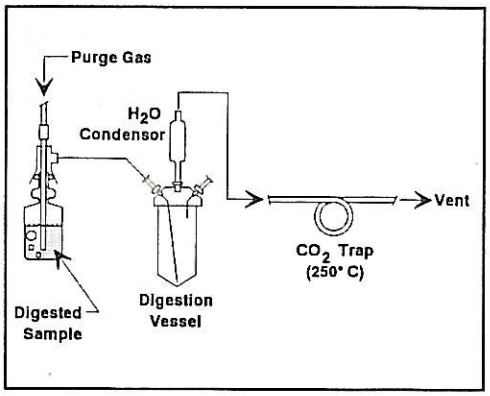


Fig. 4.28 Purging & Trapping CO_2 from Ampule

These figures show the gas flow during purging and trapping of CO_2 from the ampule into the Model 700 and...

...the subsequent CO_2 desorption and detection step.

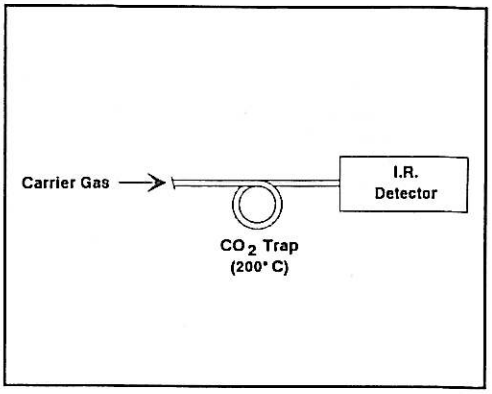


Fig. 4.29 Desorption/Detection

Operation

Follow these steps to operate in the Ampule Analysis Mode:

- Install the Ampule Capability and confirm plumbing.
- Power up.
- Enable Ampule Analysis by pressing the **SET SYSTEM CONFIGURATION** key (Ampule Analysis is Group 3 option).

Running Ampule Reagent Blanks

- Disable Auto-run by pressing the **SET SYSTEM CONFIGURATION** key.
- Press the **SELECT DISPLAY MODE** key twice to advance the Display Mode to Calibration Constants.
- Press the **SELECT NEXT DISPLAY** key repeatedly to advance the display to the OC Blank value.
- If new blank values are to be determined, set the TOC Blank to zero by pressing the **0** (zero) key then the **ENTER** key.

If the blank value in memory is not set to zero, display of subsequent millivolt values will represent the DIFFERENCE of the analyzed blank and the blank in memory. For example, a blank analysis reading of 2 mV always means that the analyzed blank is 2 mV more than the value in memory. Blank values need not be set to zero before blank determination if the analyst understands to ADD (or subtract, if negative) the average displayed value to (or from) the value in memory after blanks are determined by replicate analyses.

WARNING: Ampules may explode when crushing top. Always wear protective clothing and eye goggles.

- Mount an ampule prepared for blank determination in the ampule breaking assembly as shown in Fig. 4.30.
- Press the **CLEAR** key.
- Press the **RUN/STOP** key to start the blank run.

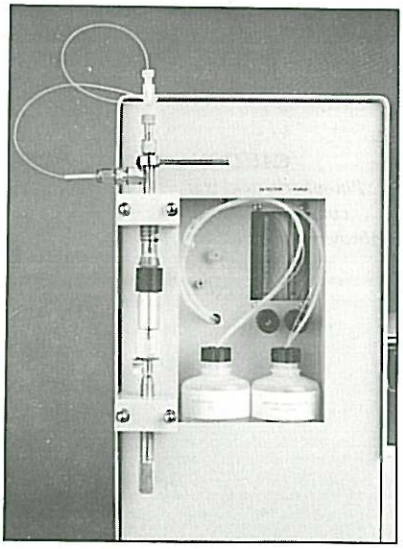


Fig. 4.30 Ampule Breaking Assembly

When the message:

BREAK AMPULE, INSERT PURGE TUBE AND "RUN"

appears on the screen, break the top of the ampule with the cutter-plunger and insert the stainless steel purge tube into the sample.

- Press **the RUN/STOP** key again to continue to analysis.
- At the end of the blank run, the detector response in millivolts will be displayed for TOC blanks on the screen, and should be recorded by the analyst unless a printer is connected to record values. The . (decimal) key may be used to recall last values.
- Allow the instrument to run replicate blanks until millivolt values are consistent.

The TOC blank millivolt values should be in the range of 20 to 30 millvolts, depending on the care taken in ampule preparation. Refer to the Ampule Purging and Sealing Manual for a discussion of preparation techniques.

- When blanks are consistent, they should be averaged and the value entered into the OC blank value (CALIBRATION CONSTANTS display mode).

After the instrument has been in operation for an extended period of time, ampule blank values may be so consistent that redetermination of blank values continually yields near 0 mV when prior blank values are left in memory. This indicates that the DIFFERENCE in the redetermined blank and the prior blank is negligible. In this case, the user may conclude that blank runs are not essential prior to analysis of each new sample set.

Calibration Using the Ampule Option

Calibration of the instrument is independent of the mode of analysis. For this reason, the instrument need not be recalibrated for ampule analysis if an accurate scaling factor is in memory. Standard carbon solutions in ampules could be used to determine the scaling factor if following one of the procedures in the previous section is not desirable. In this case, the number of micrograms carbon in the ampule and the average millivolt response from the standards should be entered into memory as outlined in previous sections.

CAUTION: Phosphoric acid is a corrosive material. Always wear protective clothing and eye goggles.

An alternative calibration method involves the use of the standarization injection vial. When the standardization vial is mounted in the cutter-plunger assembly, ca. 2 ml of 5% phosphoric acid can be used to acidify microliter injections of TIC standard (see Fig. 4.31). With the purge tube in the acid solution, press the **RUN/STOP** key.

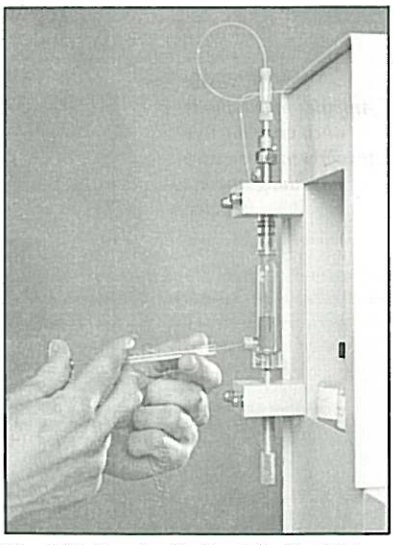


Fig. 4.31 Standardization Injection Vial

When the screen prompt instructs to:

BREAK AMPULE, INSERT PURGE TUBE AND

inject a known microgram quantity of inorganic carbon standard (i.e. standard mass) into the acid solution, then press the **RUN/STOP** key. The remainder of the analysis for the TIC standard is automatic until the end of the sequence. The average millivolt response for the standards should be entered into memory.

Running Ampule Samples for TOC

- Disable Auto-run by pressing the **SET SYSTEM CONFIGURATION** key.
- Press the **SET SAMPLE VOLUME** key and enter the number of milliliters of sample placed in the ampule.

For solid samples, enter the number of grams.

- Mount an ampule prepared for TOC determination in the ampule breaking assembly as shown in Fig. 4.30.
- Press the **CLEAR** key.
- Press the **RUN/STOP** key to start the sample run.

When the message:

BREAK AMPULE, INSERT PURGE TUBE AND

appears on the screen, break the top of the ampule with the cutter-plunger and insert the stainless steel purge tube into the sample.

WARNING: Ampules may explode when crushing top. Always wear protective clothing and eye goggles.

- Press the **RUN/STOP** key to continue the analysis.
- At the end of the analysis sequence, the detector response in millivolts and the concentration in ppm C will be displayed on the screen, and should be recorded by the analyst unless a printer is connected to record values. The . (decimal) key may be used to recall last values.

Wafertoc Analysis

Concept

A wafer is placed in the wafertoc digestion chamber and the chamber top is replaced. Acid and oxidant are introduced and the carbon present on the wafer surface is oxidized to CO_2. After a preset reaction time, the digestion chamber is placed in-line with the molecular sieve trap and a gas stream purges out any CO_2 produced by the persulfate oxidation. This CO_2 is carried to the trap held at 25°C where it is trapped and concentrated. The trap is then placed in-line with the detector and heated. The CO_2 is carried into the detector, and the resulting

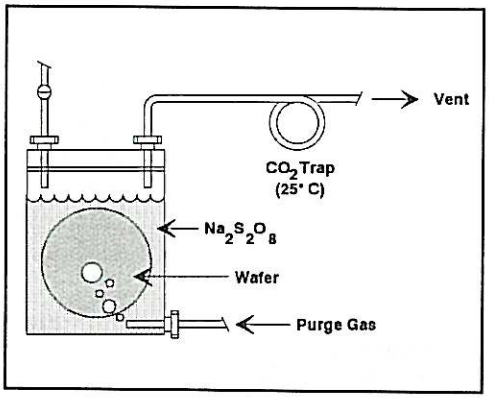


Fig. 4.32 Purging & Trapping CO_2 from Wafertoc

mass of organic carbon in the sample is displayed/printed. The expected range of carbon is 2-4 ug C for a clean wafer and up to 20 ug C for a contaminated wafer.

Operation

Follow these steps to operate in the Wafertoc Analysis Mode:

- Install the Wafertoc Capability and confirm plumbing.
- Power up.
- Enable Wafertoc Analysis by pressing the **SET SYSTEM CONFIGURATION** key (Wafertoc Analysis is a Group 3 option).

Running Reagent Blanks

- Fill the Wafertoc chamber with oxidant solution to within 1/2" (2 cm) of the top.
- Turn on Wafertoc heater block and allow temperature to stablize at 100° C ± 2° C.

It is desirable to pre-clean the wafer chamber by having allowed the persulfate digestion solution to "cook" for 1-2 hours at 100° C before actual analysis of blanks is performed.

- Enable the Ready/Standby Status Override.
- Set the digestion vessel temperature to 0°C.
- Enable Auto-run by pressing the **SET SYSTEM CONFIGURATION** key.
- Press the **SELECT DISPLAY MODE** key twice to advance the Display Mode to Calibration Constants.
- Press the **SELECT NEXT DISPLAY** key repeatedly to advance the display to the OC Blank value.
- If new blank values are to be determined, set the OC Blank to zero by pressing the **0** (zero) key then the **ENTER** key.

If the blank value in memory is not set to zero, display of subsequent millivolt values will represent the DIFFERENCE of the analyzed blank and the blank in memory. For example, a blank analysis reading of 2 mV always means that the analyzed blank is 2 mV more than the value in memory. Blank values need not be set to zero before blank determination if the analyst understands to ADD (or subtract, if negative) the average displayed value to (or from) the value in memory after blanks are determined by replicate analyses.

Typical Wafertoc blank millivolt values should be in the range of 20 to 30 millivolts.

- Press the **RUN/STOP** key to start the blank run.
- At the end of the blank run, the detector response in millivolts will be displayed for TOC blanks on the screen, and should be recorded by the analyst unless a printer is connected to record values. The . (decimal) key may be used to recall last values.

- Allow the instrument to run replicate blanks until millivolt values are consistent.

- When blanks are consistent, they should be averaged and the value entered into memory to gain the proper display and adding the average blank value to the blank value in memory for OC.

After the instrument has been in operation for an extended period of time, blank values may be so consistent that redetermination of blank values continually yields near 0 mV when prior blank values are left in memory. This indicates that the DIFFERENCE in the redetermined blank and the prior blank is negligible. In this case, the user may conclude that blank runs are not essential prior to analysis of each new sample set.

- Disable the Auto-run option to end blank cycling.

Calibration Using the Wafertoc Option

Calibration of the instrument is independent of the type of analysis. For this reason, the instrument need not be recalibrated for Wafertoc analysis if an accurate calibration scaling factor is in memory. Standard carbon solutions (microliter quantities) injected into the Wafertoc digestion chamber could be used to determine the scaling factor. To perform wafer chamber calibrations, follow the steps outlined below. However, instead of adding a wafer, inject a known amount of carbon standard (i.e. standard mass) using a microliter syringe. In this case, the number of micrograms carbon injected and the average millivolt response from the standards should be entered into memory.

Running Wafer Samples for TOC

Confirm that a representative reagent blank and a correct scaling factor have been entered into memory. Confirm Wafertoc heater temperature is 100°C ± 2°C.

It is desirable to pre-clean the wafer chamber by having allowed the persulfate digestion solution to "cook" for 1-2 hours at 100°C before actual analysis of blanks is performed.

- Confirm that WAFERTOC ANALYSIS has been enabled (Group 3, SET SYSTEM CONFIGURATION).

WARNING: The Wafertoc digestion chamber and the solution inside is extremely hot. Always wear chemical resistant, heat resistant gloves when working around Wafertoc chamber.

- Disable the sample pump, sample loop, and auto-run, options in SET SYSTEM CONFIGURATION.

- Allow any blank run to finish.

- Press the **RUN/STOP** key to begin analysis sequence. Wait for timer to stop (25 sec) and message to appear on screen.

HOT PERSULFATE is extremely corrosive and a strong oxidizer. Always wear appropriate protective clothing and eye goggles when working around Wafertoc chamber.

- Open the wafer chamber, confirm that the persulfate solution level is within 1/2" 2 cm of the top, remove any old wafer and drop a new wafer into the chamber.

- Immediately relatch the lid and press the **RUN/STOP** key.

- The carbon on the wafer surface will be oxidized to CO_2, the CO_2 will be purged from the chamber, trapped, desorbed, and detected. Results will be displayed/printed as ug C.

- When the timer stops (red lamp on RUN/STOP key quits flashing), another analysis may begin.

- At the completion of analysis, drain the Wafertoc chamber using the 50 ml syringe provided.

Serial Port Operation

Serial Output Mode Options

M0 - The serial communications port outputs the same data as the parallel printer port. Remote control of the 700 is still possible. Mode 0 is the default mode.

M1 - The serial communications port outputs the same data as the Model 700 front panel display. In mode 1 the display is updated every one second or as often as possible with the selected baud rate. An update also occurs at any display message change or when prompted with the SPACE bar.

M2 - Same as M1 except update occurs every 2 seconds.

M3 - Same as M1 except update occurs every 4 seconds.

M4 - Same as M1 except update occurs every 8 seconds.

M5 - Same as M1 except update occurs every 15 seconds.

M6 - Same as M1 except update occurs every 30 seconds.

M7 - Same as M1 except update occurs every 60 seconds.

M8 - Screen update occurs only when the display message changes or when prompted by pressing the space bar.

M9 - Screen update occurs only when the SPACE bar is pressed.

Command Code Summary

A SELECT ANALYSIS MODE
C SET SYSTEM CONFIGURATION
D SELECT DISPLAY MODE
H SET TEMPS (HEATERS)
I 10X SENSITIVITY (IR)
J PRIME ACID
K PRIME OXIDANT
L LOG ON/OFF FOR REMOTE CONTROL OF UNIT
M MODEL SELECT (SEE SERIAL MODE DESCRIPTIONS)
N SELECT NEXT DISPLAY
O OPTIONS KEY
P SAMPLE PUMP
R RAPID CLOCK ADVANCE
S SET SAMPLE VOLUME
T SET TIMES
X SET ACID VOLUME
Y SET OXIDANT VOLUME

1	ONE	(NPOC TRAP VALVE)
2	TWO	(POC TRAP VALVE)
3	THREE	(SAMPLE LOOP)
4	FOUR	(NPOC TRAP HEAT/COOL)
5	FIVE	(POC TRAP HEAT/COOL)
6	SIX	(SAMPLE DRAIN)
7	SEVEN	(ACID INJECT)
8	EIGHT	(OXIDANT INJECT)
9	NINE	(SAMPLE PURGE)

ESC	- CLEAR ENTRY
SPACE	- RE-TRANSMIT DISPLAY MESSAGE
RETURN	- ENTER
.	- DECIMAL POINT
^R	RUN/STOP
^P	PRINT
^C	CLEAR

Using A Serial Printer

The serial communications port can be used to drive a serial interface printer in place of or in combination with the parallel printer output. The instrument powers up in serial output mode **M0** which allows immediate serial printer operation, if the proper baud rate has been set on the I/O board. In serial mode **M0**, the same information that is normally sent to the parallel printer output will be echoed to the serial printer port.

Using A Remote Terminal

The Model 700 can be connected to remote terminal to allow total remote control and data monitoring. Connection between the instrument and the terminal can be through direct 3-wire connection, a fiber optic RS-232 link, or through telephone lines using a MODEM at each end. Instructions for operation are:

This description assumes that a direct connection is being used or the telephone connection has already been established.

- Confirm proper construction of the serial cabling according to the particular application and equipment used. Note that only three wires are required for proper operation: signal ground, transmit, and receive. No handshaking lines are required or used.

- Confirm connection of the cable between the Serial Communications connector on the rear of the instrument and the terminal/modem/etc.

- Confirm setting of desired baud rate using the DIP switches on the I/O board to match the baud rate of the connected device. Normally 300 or 1200 baud will be selected if the instrument is connected to a modem. If a direct wire connection is used, up to 9600 baud may be used depending on cable length. Baud rates slower than 300 are not recommended because they will slow down the print rate of the parallel printer when operating in serial mode M0.

- The setup should now be complete to allow remote control and monitoring of the instrument. To test the setup, turn on power to the instrument and terminal.

- Press the **PRINT** button on the instrument control panel. The terminal screen should begin to display the same information that normally is printed on the parallel printer when it is connected.

- If this does not occur, verify the setting of the baud rate switches on the instrument and on the connected device, and make sure that they are the same. Also check the cable connections and make sure that Transmit on the Model 700 is connected to Receive on the terminal and that Receive on the Model 700 is connected to Transmit on the terminal.

Remote Operation Using Modem

- Long distance remote control and monitoring can be accomplished by connecting the instrument to an "Auto-answer" modem.
- Dial the phone number of the instrument and wait for the modem to answer and connect.
- When the carrier from that modem is heard or detected, turn on or connect the modem at the terminal end.

The baud rates must be compatible for proper operation.

- Press **L** on the terminal to log onto the instrument.
- Type **M1** to start the instrument transmitting the current display information. This verifies that bi-directional communication has been established.
- Now proceed as if there were a direct wire connection between the instrument and the terminal.
- When done communicating with the instrument, log back off by pressing **L** a second time. Logging off of the instrument helps protect against random serial data from accidentally being interpreted as remote control codes.

Sample Set - Key Sequence

The following is a sample set of terminal key sequences to log-on or establish contact with the instrument.

Key	Keyboard Equivalent	Comments
L	None	Log onto the instrument
M2	None	Set to Mode 2, display should update once every two seconds
C	System Configuration	Enter system configuration
RET	Enter	Step through list
RET	Enter	Continue stepping through. Enter 1 or 0 to enable/disable flags
RET		Enter Continue until done with configuration

Key	Keyboard Equivalent	Comments
ESC	Clear Entry	Abort system configuration. Return to Main display
A	Select Analysis Mode	POC/TIC/TOC analysis display
A	Select Analysis Mode	Calibrate mode display
MB	None	Select Mode 8, Display update stops
N	Select Next Display	Step through and verify blank
N	Select Next Display	Continue until all values are shown
A	Select Analysis Mode	Now in manual mode. Diagnostics display now shown
N	Select Next Display	Step through and view temps, valve press until H1/H2 temp display is shown
N	Select Next Display	Step through andview temps, valve press until H1/H2 temp display is shown
M1	None	Display will update once per sec
4	4 (H1 Heat/Cool)	Manual control of H1, NPOC trap heat. See temperature change
1	1 (NPOC Trap Valve)	Manual control of NPOC trap valve
6	6 (Spl Drain)	Toggle sample drain valve
C	Clear	Reset instrument to standby state
A	Select Analysis Mode	Back to TIC/TOC analysis mode
M4	None	Update display every 8 seconds
S	Set Sample Volume	View current setting
5.25	5.25	Set to 5.25 ml sample volume
Space	None	Update display to verify value
Esc	Clear Entry	Recall old value, clear new entry
Space	None	Update screen
5.15	5.15	Enter desired sample volume

Key	Keyboard Equivalent	Comments
Space	None	Verify numbers
Ret.	Enter	Enter new sample volume
P	Print	Print out current presets
R	Run/Stop	Start analysis cycle
M8	None	Updates only on message change. Can let run in this mode to monitor whole analysis
M0	None	Screen will only show analysis results (same as printer)
L	None	Log off of instrument. Erroneous serial data is now ignored by 700
L	None	Toggle log off/on back to on. Remote control is again possible
C	Clear	Stop timer and abort analysis

Standby/Power Down

Although the Model 700 is designed for continuous operation, power down overnight or for longer periods between analyses is recommended for gas conservation. If gas conservation is not a consideration, the instrument may be left in the READY condition with gas flowing, and may be left continually cycling reagent blanks, or may be left continually cycling clean water for cleanup purposes. The user may determine by experience which of these options is most suitable, depending on the sample concentration level, number of samples routinely analyzed, and time period between analysis sets. Procedures for each of these options are as follows:

Clean Water Cycling

Repetitive analysis of reagent water is the best method for continual cleanup of the Model 700. Reagent water cycling is used as a method for instrument cleanup after assembly during the checkout stage of instrument production, and is recommended before attempting analysis of low level water samples. Follow these steps to cycle with water.

A delay time between analyses may be entered using the SET TIMES key to conserve water and reagents.

- Confirm that there are sufficient reagents, printer paper, and gas for repetitive cycling during the standby time period.
- If the Process Sampling Capability is attached, install a 5 ml sample loop.
- Enable the SAMPLE PUMP, SAMPLE LOOP and AUTO-RUN options.
- Set SAMPLE VOLUME to 5 ml.

A sample volume setting of 5 ml will allow 11 ml of sample to be pumped per cycle.

- Set ACID VOLUME to 500 ul and OXIDANT VOLUME to 4500 ul.
- Place the loop sampling tube into a volume of reagent water suitable for repetitive cycling during the standby period.
- Confirm that the volume of the waste receptacle is sufficient for repetitive cycling during the standby period.
- Press the **RUN/STOP** key to start the repetitive cycling.

Reagent Blank Cycling

Repetitive analysis of reagent blanks during standby allows the instrument to be used at any time for immediate analysis of samples. If a printer is connected, blank values may be constantly monitored and corrected if needed. Follow these steps to cycle blanks.

- Confirm that there are sufficient reagents, printer paper, and gas for repetitive cycling during the standby time period.
- Follow the steps outlined in Running Reagent Blanks earlier in this chapter.

Standby in READY Condition

The instrument may be left on indefinitely with gas flowing without harm. Analyses may be performed after this standby period without needing to wait for the IR, digestion vessel, or POC furnace to warm up. Follow these steps:

- Confirm that there is sufficient gas for continual flow during the standby period.
- At the end of the standby period, press the **CLEAR** key to bake the primary trap before analysis.

Power Down

To conserve gas, the instrument may be powered down on a daily basis without affecting the calibration data and other values in memory. A 10 minute warm-up period is necessary before analysis on subsequent power-up. To power down, follow these steps:

- Press the **CLEAR** key to insure proper valve positions upon power down.
- Turn off the MAIN POWER switch.
- Turn off power to any optional equipment not plugged into the Model 700.
- Turn off the gas flow to the instrument.

Analytical Hints

Standard Additions

A commonly used method in calibrating instruments involves the addition of a known standard to an unknown. By analyzing the unknown sample and a standard added to the unknown, the instrument's detection of the sample can be calculated. The difference between the instrument's response due to the standard plus unknown and the standard's true value is then used to adjust (or calibrate) the instrument. After the instrument is calibrated to the corrected value the unknown can be analyzed directly.

The standard addition calibration is often used when analyzing TOC in ultra-pure water. The term, TOC, is used in this application to mean Total Oxidizable Carbon, that is, those organic species which can be oxidized to carbon dioxide (CO_2) by a given oxidation method. Analyzing the oxidizable species in ultra-pure water bears special consideration due to the low concentration of these species and to the fact that this same water is used to set the baseline and make the standards. Dealing within the range of 0-1000 ppb, the calibration method is quite important. The standard addition method is able to meet this demand for precision and accuracy. To expand this theoretical method into an on-hands application, a Model 700 was calibrated with ultra-pure water and a 1 ppm KHP standard spike. The standard addition sample is viewed as a standard spike since it is a known standard added to an unknown. The example that follows is a calibration for TOC. This same calibration procedure can be applied to TIC but it should be noted that CO_2 is absorbed rapidly in ultra-pure water, and at this low level of detection even the smallest amount of CO_2 can cause considerable inaccuracies in the TIC values.

Reagent Blanks

In order to negate the interferences due to reagents, gases, etc., reagent blanks are run.

- Set blank (IC and OC) mV values to zero
- Set: Standard mass to 1.00 mg C,
  Standard average to 1.00 mV, and
  Scaling factor to 1.00 mgC/mV
- Run Blanks as described in the section Running Reagent Blanks
- Collect three good repetitions, TIC and TOC mV values.

Example:
TIC = 0.88548 mV
TOC = 3.14908 mV

TIC = 0.88548 mV
TOC = 3/14908 mV

TIC = 0.85689 mV
TOC = 3.26396 mV

- Calculate the average and enter as blank mV values.

Average IC = 0.87595 mV
Average OC = 3.18737 mV

Calibration

The low levels of oxidizable carbon involved in ultra-pure water calls for the practicality of a 1 ppm KHP standard spike and the use of a 10 ml loop for highest precision.

- Prepare calibration standard spike
- 1 ppm KHP standard spike

The water used to make the spike should be from the same source and obtained at the same time as the ultra-pure reagent water that will be analyzed. This will decrease the water inconsistencies due to organic contamination.

- Run Calibration

Run ultra-pure, reagent water. At this point the mV values obtained are the only accurate measurements of detector response. The ug C and ppm values can, for all practical purposes, be ignored until after the calibration is complete.

- Collect three good repetitions. Example:
  TOC = 6.12489 mV
  TOC = 6.06676 mV
  TOC = 6.00864 mV
- Calculate the average: TOC = 6.067 mV
- Run 1 ppm C KHP standard spike
- Collect three good repetitions. Example:
  TOC = 198.277 mV
  TOC = 198.157 mV
  TOC = 199.968 mV
- Calculate the average: TOC = 198.801 mV

Calibration Calculations

Standard mass (ug C) = standard spike concentration x loop size. For this experiment a 10.483 cc loop was used, therefore:

Standard mass = 1 ppm C x 10.483 cc = 10.483 ug C

Standard average = (avg. std. spike mV response) - (avg. ultra-pure reagent water mV response) = 198.801 mV - 6.067 mV = 192.734 mV

- Set calculated values into the calibration constants

The scaling factor is automatically calculated to be 0.05439 ug C/mV

Accuracy Verification

- Rerun 1 ppm KHP standard spike
- Collect two good repetitions. Example:

TOC = 198.961 mV	10.8217 ug C	1.03231 ppm C
TOC = 199.766 mV	10.8655 ug C	1.03649 ppm C

- Average the ppm C value: TOC 1.0344 ppm C

From this value, 1.0 ppm corresponds to the standard spike and the remaining 0.0344 ppm corresponds to the ultra-pure reagent water used to make the standard spike. This can be verified by analyzing the ultrapure reagent water again.

Rerun Ultra-pure Reagent Water

- Collect three good repetitions. Example:

TOC = 6.47378 mV	0.35211 ug C	0.03359 ppm C
TOC = 6.55305 mV	0.35642 ug C	0.03400 ppm C
TOC = 6.58470 mV	0.35814 ug C	0.03416 ppm C

- Average the ppm C values. Example: TOC = 0.0339 ppm C

This value, 0.033 - 0.034 ppm C, is the correct TOC measurement for the ultra-pure reagent water.

Discussion

In calibrating, it is very important to remember to subtract the ultra-pure reagent water mV average from the standard spike mV average. If this value is not subtracted, the standard average will be incorrect. For example, with the correct standard average of 192.734 mV and standard mass of 10.483 ug C, the scaling factor is 0.05439 ug C/ mV. This KHP standard spike gave a value of 1.03649 ppm C.

If, however, an incorrect standard average of 198.801 mV (by not subtracting the reagent water response) and the same standard mass were used, the scaling factor would be 0.05273 ug C/mV. This scaling factor applied to the 199.766 mV response of the 1 ppm C KHP standard spike would yield 1.00486 ppm C. This ppm C would have implied that ultra-pure reagent water was 0.00486 ppm C, (once the 1 ppm C corresponding to standard spike is eliminated.)

The method of standard addition is useful not only in calibration of instruments but also for verification of recoveries in any analytical analysis. It is especially useful when accuracy is questionable at low concentration and a separate confirmation analysis is not feasible.

Samples Containing Brine or HCl

High levels of chloride ion in TOC samples present special problems for analysis. The major factors affecting successful analysis are maintaining effective sample/oxidant ratios, allowing sufficient analysis time, and reducing the corrosive effects of the chlorine produced.

Chloride ion competes with carbon for available persulfate ion. A typical brine or HCl sample may have chloride levels 1000-10,000 x that of the organic carbon concentration, so, without precautions, poor recovery of TOC may be expected due to incomplete oxidation.

The oxidation of organic compounds by persulfate generally follows first order reaction kinetics. The oxidation of chloride to chlorine introduces intermediate steps, resulting in a more complex reaction. This reaction proceeds more slowly, and organics may not be completely oxidized during the normal reaction time.

The chlorine produced in the oxidation of a brine or HCl sample may cause corrosion of the molecular sieve trap and may affect the ability of the molecular sieve substrate to efficiently trap and release CO_2.

The negative results of high chloride levels can be partially offset by use of sample volumes of less than 100 ul and dilution. The microliter sample loop capability (OI Part #172-784) is recommended. Analysis of concentrated hydrochloric acid (36-38%) is not advised. Concentrated acid should be diluted 50:50.

- Remove the acid outlet fitting from the top of the syringe injection port and replace with a 1/4"-28 plug (OI #166-430).
- Install the Kel-F tee (OI Part #170-431) between port #1, valve C and port #3, valve B. Install acid outlet fitting in remaining port of the tee.
- Place the 100 ul or 40 ul loop directly into ports #3 and #6 of valve C. Set the appropriate volume by pressing the select sample volume key and entering the volume.
- Set acid volume to 400 ul. Set oxidant volume to 5000 ul.
- Set extended reaction time to 6 minutes.
- Select calibrate mode by pressing the **Select Display Mode** key.
- Set OC blank to zero.
- Set instrument to run blanks and run reps to establish new OC blanks. Enter new value.
- Enable sample loop and sample pump and run samples. Oxidation efficiency may be verified by spike recovery of a chloride sample or standard (i.e. Standard Addition).

At the conclusion of chloride sample analysis, rinse system with a large volume of distilled or deionized water to remove any acid residues from line and pump.

Confirming Recoveries in Difficult Samples

The default analysis conditions set in the Model 700 are times, temperatures and volumes that have been established to analyze the majority of water samples typically tested in laboratories. However, in the case of complex sample matrices, these conditions may not be adequate to render an accurate value for carbon concentrations in a sample.

There are several methods for determing recovering efficiency on a sample. One discussed earlier in this chapter, Standards Addition, is probably the most common technique used. Another would involve using a smaller or larger sample size and varifying that concentration (recovery) is equivalent in both cases. The method described here is used to not only give information on method recoveries but when completed, sets the conditions for analysis on the sample matrix in question. In brief, it requires the analyst to vary the instruments analysis parameters until a maximum, stable value is obtained.

There are three parameters that can be adjusted to achieve optium analysis conditions. These are (1) temperature of heated zones which included the digestion vessel and the POC furnace (for Purgeables analysis), (2) volumes of reagents used which are the phosphoric acid (TIC), persulfate (TOC) and (3) times allowed for purging inorganic carbon (extended purge) and conversion of organic carbon to CO_2 (extended reaction). For the most part, the first two parameters seldom need adjusting. Especially the 100° C set point for the digestion vessel since lowering the temperature much below 100°C starts slowing down reaction rates and elevating the temperature causes

excessive steam generation which can result in problems downstream of the reaction vessel. Increasing the reagent volumes may be necessary if samples have a high pH, particulated inorganics or if carryover from one sample to the next is suspected. So, for the majority of difficult samples (brines, acids, caustics, SOC, etc.) the parameters that achieve the most significant changes are the extended purge and extended reaction parameters.

To confirm that the time parameters are at an optimum, the analyst should choose his/her suspected, "most difficult" sample to work up the analysis conditions. Beginning with default analysis parameters, run this sample two to three times to establish a trend. Then extend the time function that is believed to be too low using 30 sec to 1 minute increments. That is, if low TIC recoveries are suspected or if it is believed that inorganic carbon is being carried over into the TOC, then extend the purge time. If TOC recoveries are lower than expected, or they are not reproducible, extend the reaction time. Low POC recoveries are handled in the same way as low TIC with one exception; the standard trap used with the Model 700 is packed with Tenax GC which has a limited retention time for some of the lighter purgeable gases (freon 12 for example). Thus, extending the purge too long can result in these species breaking through the POC trap, therefore going undetected.

Optimum analysis time has been achieved when extending the time parameter in question has no significant increase in IR detector response (greater than 10 mV). This increased response can be monitored in two ways. Either by monitoring the 700 output printer or by connecting a strip chart recorder, set at 1 volt full scale output, to the IR output connections located on the back, upper righthand corner of the instrument. Remember that changing the analysis conditions will have some effect on the blank value which will increase slightly with increased time or reagent values.

Once new analysis conditions have been determined on the "most difficult" samples, the instrument can be calibrated and the other "less difficult" samples can be analyzed using the analysis method established.

Chapter 5
Maintenance

In the last chapter, an overview of the methods used by the 700 for sample analysis were given, followed by step-by-step instructions for power up, sample introduction, running blanks, and running various types of samples. This chapter discusses both the routine and non-scheduled maintenance of the 700, starting with some general information and a maintenance schedule.

The following terms are used throughout this manual in reference to components of the Model 700.

General Information

Definitions

1/8" TFE Tubing: 1/8" OD x 0.062" ID TFE teflon tubing (OI Part #147-901).

1/16" TFE Tubing: 1/16" OD x 0.031" ID TFE teflon tubing(OI Part #145-591).

A procedure for flaring the 1/8" and 1/16" tube end fittings is outlined later in this chapter .

1/8" Tube End Fitting: Polypropylene Fittings with 1/4" x 28 threads on one end and a 5/16" hex or square head on the other. The 1/8" end fittings have a 1/8" hole through them to be used with 1/8" TFE tubing. The tubing is to be flared to keep the end fitting and accompanying steel washer from sliding off. These are stocked in several different colors, each with washer. OI Part #s are:

G - Green - 166-357
B - Blue - 166-381
R - Red - 166-365
Y - Yellow - 166-373
K - Black - 166-399
L - Natural - 165-862

1/16" Tube End Fitting: Polypropylene fittings with 1/4" x 28 threads on one end and a 5/16" hex or square head on the other. The 1/16" end fittings have a 1/16" hole through them to be used with 1/16" TFE tubing. The tubing is to be flared to keep the end fitting and accompanying steel washer from sliding off. These are stocked in several different colors, each with washer. OI Part #s are:

G - Green - 166-290
B - Blue - 166-331
R - Red - 166-307
Y - Yellow - 166-315
K - Black - 166-323
L - Natural - 166-282

Coupling: a nylon coupling for tube end fittings with an internal 1/4" x 28 thread (OI Part #166-274)

Valve (A to D) Port (1 to 6): there are two, three, or four 6-port valves used on the system, depending on options. The valves are teflon with six teflon tubes protruding from each. The valves are attached to large black and silver valve actuators. The six

ports on each valve are marked 1 through 6. We designate the valves A through D (See Figure 7.4.1). Valves A and B are mounted directly to the vertical chassis (A is in rear). Valves C and D, if used, are mounted on brackets attached to the horizontal shelf (D is in rear). Viewed from above, the valves would be in these relative positions:

	Rear	Front
Innermost	A-Primary Trap Valve	B-Drain Valve
Outermost	D-POC Trap Valve	C-Sample Valve

Valves A and B are used on all units. Valve C is used only with the process sampling option. Valve D is used only with the purgeables capability option. Units with both options use all four valves.

Plumbing instructions will often refer to a specific port on a 6-port valve such as Valve B Port 4. Valve plumbing is somewhat different for units with the two options. Separate drawings for each combination are given in this manual in **Chapter 9**.

Making 1/8" and 1/16" Flare Fittings with TFE Tubing

- A 40W soldering iron (OI Part #168-808) with stainless steel flaring tips is used to thermally flare TFE tubes. A 1/8" tip (OI Part #169-129) and 1/16" tip (OI Part #169-137) are required. These tools are included in an accessories kit (OI Part #168816). Directions for flaring tubing are as follows.
- Make sure the tube end to be flared is cut squarely at 90°.
- Slide the appropriate tube end fitting and washer onto tube. Allow 1/2" between washer and end of tube to avoid preheating washer.
- Use a "Kimwipe" or sandpaper to grip the head of the tube end fitting and tubing. Press the tubing over the tip of the soldering iron. Apply pressure until the tip of tubing flares out.
- Slide the end fitting and washer firmly against the flare. Hold for approximately 1/2 second and immediately remove and press flare against a flat, cool surface.
- Inspect the flare for uniformity, diameter, cracks or other defects. 1/8" TFE tubing should flare to a diameter slightly larger than the stainless steel washer. 1/16" TFE tubing should flare to approximately 1/8" diameter in order to hold onto the washer.

Schedule for Routine Maintenance

For the most reliable performance of the instrument, and as a condition of the warranty, the following schedule of routine maintenance should be followed. (Scheduled hours refer to number of hours of operation)

The user is encouraged to setup an instrument log book to record instrument operation time and document periodic maintenance. This log book can be used to record results of inspections and component replacement necessary for proper maintenance of the instrument.

Maintenance Item	Schedule
Reagent Reservoirs	25 to 2500 Injections
Injection Port Septum	50 to 200 Injections
Tube End Fitting Connections	100 hours
IR Zero	100 hours
Gas Service	100 hours
Six-Port Valves depending on application	200-2000 hours
Sample Pump	2000 hours
Activated Carbon Scrubber	2000 hours
Digestion Vessel/ Condensation Chamber	2000 hours
Permeation Tube	2000 hours
IR Cell depending on application	2000-4000 hours
IR Linearization	User determined

Reagent Reservoir Maintenance

The volumes of reagents in the bottles on the front of the instrument should be inspected at times according to the number of samples run and volumes of reagents used per sample. The reagent bottles hold 250 ml each. Reagents should be added to keep the bottles from being completely emptied. Operation of the reagent pumps without liquid is not recommended. See **Chapter 1** for the preparation of reagents for addition to these bottles.

Changing Injection Port Septum

Behind the injection port is a 5 mm teflon faced, silicone rubber septum (OI Part #166-133). This septum is pierced when samples are injected by syringe. Depending on the quality of the syringe needle, this septum may need to be replaced after 50 to 200 piercings. A water droplet coming from the injection port hole during sample-draining (near the end of each run) is an indication of a leaky septum. Though a leaky septum is cause for replacement, no sample loss will occur during injection, so the septum need not be replaced until a convenient time. Water which seeps from the injection port in this manner is the spent sample normally drained to waste under gas pressure. Change the septum as follows:

- Insert a syringe needle (2" long) through the injection port guide.
- With the syringe needle in the injection port guide, unscrew the guide counter-clockwise.

When the injection port guide clears the final threads of the injection port block, remove the syringe with the guide and septum on the needle.

- Discard the spent septum and place a new septum on the syringe needle, centered with respect to the injection port guide and with teflon face away from injection port guide.

- Install the new septum into the injection port block by screwing the injection port guide clockwise into the injection block until it just becomes finger-tight. **DO NOT OVERTIGHTEN.**

- Check the injection port for leaks.

Tube End Fittings

The tube end fittings used to interconnect the valves, digestion vessel and various other connections in the Model 700 are subject to leaks over a period of time due to "cold flow" of the teflon tube flare. At every 100 hours of operation these fittings should be checked by confirming that all tube end fitting connections are fingertight. It is also advisable that at this time a purge gas and carrier gas leak check be performed. These leak checks are described later in this chapter.

IR Zero

If IR baseline cannot be adjusted to the desired range, then problems other than zero offset are likely. Refer to IR troubleshooting in Chapter 6.

The IR detector zero (baseline) will fluctuate up or down during periods of non-use. This is due to environmental factors such as operating temperature, how long the IR case purge has been on (to expel ambient CO_2), or purity of gases (especially if oxygen is being used in the case of POC analysis). However, under routine operating conditions, the baseline millivolt reading should be set between 5 and 10 mV for optimum range and linearity response. This adjustment should be checked after every 100 hours of operation which corresponds to gas service maintenance. Procedure is as follows:

- When instrument is in READY state, remove the right bay cover to gain access to IR detector adjustment knobs.

- Press the **SELECT DISPLAY MODE** key to select IR OUTPUT.

WARNING: Do not make adjustments with SPAN control knob as this would cause problems effecting the IR detector linearity.

- Turn ZERO adjustment knob clockwise to increase baseline (positive shift) or counterclockwise to decrease baseline (negative shift) and set output between 5 and 10 mV.

- Allow instrument to perform several automated analysis and re-check baseline with instrument in READY state. Make any adjustments if necessary as described above.

- Replace right bay cover.

Gas Service

Gas consumption is listed in **Chapter 1**. Standard 2000 psi gas cylinders hold over 200 cubic feet. There are 28.32 liters per cubic foot. Thus, a standard cylinder should last at least 130 hours. Cylinder gas pressure should be monitored after each 100 hours of operation with gas flow to confirm sufficient gas for planned operation.

Six-Port Valve Maintenance

A basic system uses two 6-port rotary valves. Units with options may use one or two additional valves. These valves have fluorocarbon polymer (TFE and FEP) liquid contact parts which are chemically compatible with essentially all solvents, acids, and bases.

The valves have flat Kel-F rotors that bear against a teflon stator. Rotation is through a 60° angle. Teflon tubes attached to the valves have 1/4" x 28 tube end fittings. The valves are pressure rated at 300 psi (2070 kPa).

These valves should be inspected as frequent as every 200 hours for instruments analyzing water matrixes containing particulates or salts containing chloride. For instruments being used to analyze clean waters, such as potable waters or ultra-pure waters, the inspection of these valves can be performed every 2000 hours (based on performance of valves currently being used for these applications).

Maintenance of these valves should include cleaning and/or relapping (facing) and/or replacement if necessary. The procedure for each valve is identical as follows.

It is suggested that valves be inspected one at a time so that assembled valves can be used as a reference and leak checks can be performed on each valve once it is reassembled.

- Remove the left bay cowling to access the valves.
- Refer to Fig. 5.1. Un screw and remove the three hold down nuts and lock washers from the bottom of the mounting flange with a 1/4" wrench.
- Unscrew and remove the three Phillips head screws which hold the valve to gether. The valve need not be removed from its actuator.
- Lift the stainless steel cap with tubing up from the valve to expose the stator.

Stators have a raised surface on one side which should be placed against the valve rotor.

- Lift the stator up from the valve body by sliding it off of the two stator alignment pins. Take care to note its orientation on the valve.

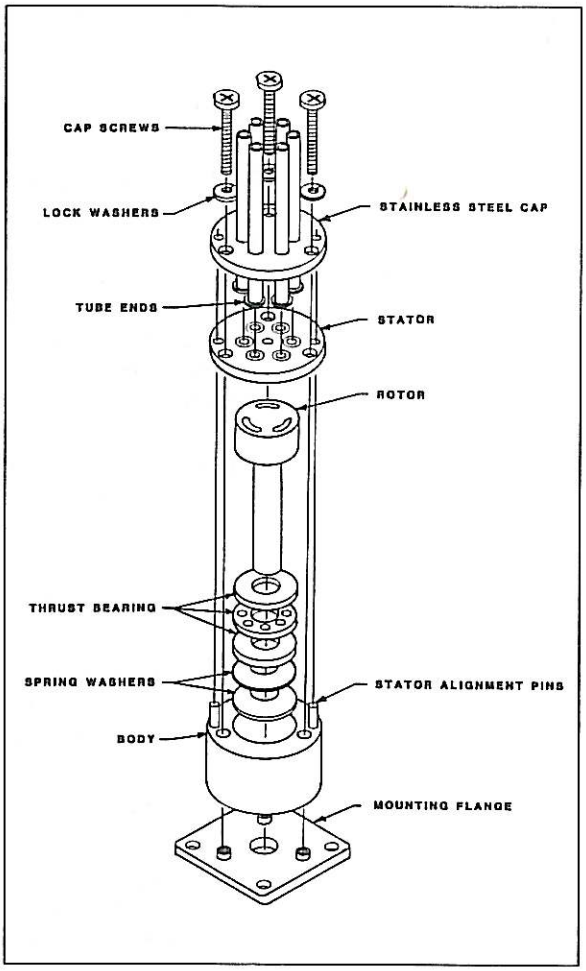


Fig. 5.1 Six-Port Rotary Valve

- Inspect the stator, tube ends, and rotor for accumulation of solid material. The six hexagonally-placed holes in the stator and the three curved grooves in the rotor should be cleaned of any deposits.
- Inspect the contact area between the stator (OI Part #172-700 for 1/8" or

#172-750 for 1/16") and rotor for signs of scratches, gouges, or abrasions. Heavily gouged or scratched components should be replaced. Light scratches may be removed by relapping with 400 and 600 grit abrasive paper, using water while lapping.

- Confirm that the tube flares which press against the stator are in good repair. If tube ends need to be reflared, refer to the previous section on making flare fittings with TFE tubing.

- Reassemble the valve by replacing the parts exactly as they were previously oriented. Refer to Fig. 5.1 for proper orientation during assembly.

Lock washers on the screws are essential for extended operation to prevent leakage. Be sure to replace them onto the three Phillips head cap screws during reassembly

- Tighten the three Phillips head cap screws uniformly with just enough pull so that the body, stator, and cap go together with only a slight uniform gap between the cap and stator (refer to an adjacent assembled valve). Do not make final adjustment at this time.

WARNING: Overtightening the head cap screws will cause stator deformation and damage to valve rotor.

- Perform a Purge Gas leak check as outlined later in this chapter and uniformly tighten the three Phillips screws until the valve just stops leaking as indicated by purge gas rotometer on front left panel of the instrument.

- Replace lock washer and hold down nuts to cap screws on bottom of mounting flange. Tighten until just snug with a 1/4" wrench.

Sample Pump Housing Maintenance

This procedure applies only to instruments with the Process Sampling Option (OI Part #164-559). This option includes a peristaltic pump mounted inside the left bay. It is used to aspirate samples through the loop sampling inlet and the sample loop (see **Chapter 9** for plumbing schematics). The pump housing consists of a length of silicone tubing (OI Part #177-247) mounted in a plastic housing. The silicone tube is considered expendible because the tubing will eventually wear out.

The tubing should be inspected after every 2000 hours of operation. More frequent inspections may be necessary if running samples containing strong acids or bases.

- Remove the plastic barb fitting from end of outlet leg (closest to outside of left bay) of the silicone tube.

- With a small, flat blade screwdriver, carefully pry apart the teeth of the plastic retaining clamp on the outlet leg of the silicone tube and remove the retaining clamp from the end of the tubing.

- Locate the slotted pump shaft in the center of the pump housing.

- With an appropriate screwdriver in one hand, turn the pump shaft counterclockwise while pulling the sample inlet leg with the other hand to remove the silicone tubing from the pump housing.

- Inspect the tubing for excessive wear, holes or cracks, and replace if these signs are evident. If the outside of the silicone tube is dry or a replacement tube is being installed, lightly coat the outside wall that will be exposed to the pump housing with a silicone grease lubricant. Installation of the tubing into the pump housing is the reverse of the above procedure.

Activated Carbon Scrubber Maintenance

An Activated Carbon Scrubber (OI Part #160-375) is plumbed in-line with the purge gas downstream of the flowmeter. It is used to trap and filter any organic contaminants from the purge gas line so they will not be oxidized in the digestion vessel and trapped during sample purge. Depending on the quality of gas used with the unit, organic contaminants may eventually break through the scrubber and cause an increase in TOC blank. In this case, or after every 1000 hours of operation (depending on purity of gas), the scrubber should be baked out or replaced.

To bake out the scrubber:

- Remove the scrubber from the unit, being carefull not to break the barbed fittings on the plastic check valve, and connect it to an inert gas line (nitrogen or helium) using a metal fitting.
- Bake the scrubber only, at 400-600° C in a muffle furnace or similar heating device under gas flow for 30 minutes, or at 300° C overnight.
- Cool the scrubber and replace it in the unit.

Digestion Vessel and Condensation Chamber Maintenance

Maintenance of the Digestion Vessel and Condensation Chamber assembly should include the inspection/replacement of the three red silicone O-rings used to seal the vessel cap. Inspection should be performed after every 2000 hours of operation to check for signs of degredation of the silicone rubber as indicated by pitting or chalky spots, and should be replaced to prevent potential leaks. This inspection may also include verifying the integrity of the two flare junction seals on the side ports of the vessel cap against leaks.

The correct assembly of the digestion vessel is shown in fig. 5.2.

Procedure for assembly is as follows:

- The short, flared teflon tube (OI Part #180-208) for directing oxidant into the bottom of the Digestion Vessel should set flush in the bottom of the yellow side port. It should extend down through the Digestion Vessel cap (OI

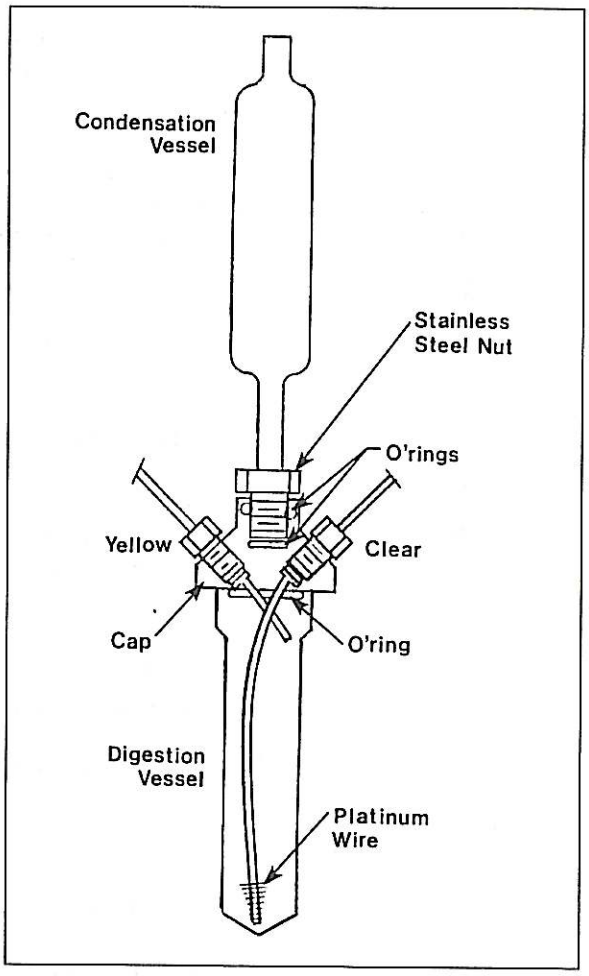


Fig. 5.2 Digestion Vessel

Part #193-557) so that it just touches the inside wall of the cap.

Positioning this tube in the very bottom of the cone is extremely critical for proper draining of spent samples. It is suggested that several short pieces be cut (at an approximate 65° angle) and then tested for proper fit, rather than attempting to measure and cut one final length.

- The long, flared teflon tube (OI Part #180-216) for sample injection, purging and draining is to be placed in a similar manner through the other side port (clear) of the Digestion Vessel cap. Upon initial installation only, this tube will need to be trimmed to a length such that when the Digestion Vessel cap and chamber (OI Part #166-076) are screwed together (with clear tube fitting and large silicone O-ring in place) the tube just touches the center of the conical bottom without bending in the Digestion Chamber.

- Once the drain tube is cut to its final length, coil the platinum, wire (OI Part #165-581) around the bottom 1/8" (3mm) of the tube.

- If the large silicone O-ring (OI Part #166-175) which seals the cap and chamber shows any signs of degradation, replace it by cutting off the old O-ring with a sharp razor or knife and then carefully working the new O-ring over the threaded neck of the Digestion Vessel cap.

- While holding the cap horizontally so that the platinum wire coil does not slide off, screw the Digestion Vessel chamber into the cap until the O-ring seals the two parts together.

- If the primary or secondary, 1/4" I.D. silicone O-rings (OI Part #192-047) on the inlet stem of the condensation chamber show any signs of degradation, replacement is required. The primary sealing)-ring is removed by simply sliding it off the end of the glass condensor. The secondary sealing O-ring is removed by first sliding the 1/4" polypropylene spacer (OI Part # 192-054) off of the glass condensor and then removing the O-ring.

- Installation of new O-rings on the Condensation Chamber (OI Part #192-039) is reverse of removal with the primary sealing O-ring positioned at the end of the condensor tube inlet stem.

The inlet stem of the Condensation Chamber is indentified as the longer (1.6") length of the two 1/4" O.D. stems.

- Insert the inlet of the Condensation Chamber into the top of the Digestion Vessel cap and carefully screw the 1/4" stainless stell nut (OI Part #178-574) down into the cap, 1/8 turn past finger-tight.

Do not overtighten as damage may occur to the glass condensor and teflon parts.

- If the 1/4" x 1/8" reducing union (OI Part #124-735) was removed from the outlet of the Condensation Chamber to facilitate the maintenance procedure, confirm that the 1/4" teflon ferrule (OI Part #175-978) is installed with the flange seated in the body of the union and tighten 1/8 - 1/4 turn (max.) past finger-tight. Reconnect the inlet of the permeation tube to the unioon and tighten 1/4 turn past finger-tight.

- Screw the yellow tube fitting from the oxidant pump into the appropriate cap port and tighten finger-tight.

- Screw the clear fitting from the injection block into the drain line port and tighten finger-tight.

- Confirm proper final position of the drain tube by visual inspection through the digestion chamber.

- Perform a purge gas leak test as outlined later in this chapter and correct leaks as necessary.

- If available, apply a thin coating of white silicone heatsink compound to the outside wall of the Digestion Chamber.

- The Digestion Vessel/Condensation Chamber assembly is now ready to be reinstalled in the heater block.

Gas Permeation Tube Maintenance

A gas permeation tube (OI Part #189-829) is plumbed between effluent of the Condensation Chamber and Valve A Port 1 (Primary Trap Valve).

This is a coaxial tube set containing a hydroscopic, ion exchange membrane in a continuous drying process to selectively remove water vapor from mixed gas streams. The membrane is a proprietary extrudible dessicant in tubular form inside an outer tube shell. When an intermittently wet gas stream flows through the inner tube while a dry gas purges the shell in a counter-current fashion, water vapor molecules are transferred through the walls of the tubing.

The ion exchange membrane is chemically resistant to all gases and liquids. However, the drying capacity may be decreased if it becomes contaminated with non-volatile liquids or salts. For this reason, the permeation tube should be cleaned or replaced after every 2000 hours of operation as preventative maintenance.

Never remove or loosen any part of the tee fittings on the permeation tube except the nuts on each side arm.

Inorganic salts that are adsorbed into the membrane can be removed by 10% nitric acid at 50-60° C as follows:

- The Tube, with tee fittings intact, should first be removed from the system.

- Remove the gas/liquid separator disc (OI Part #192-120) from the barb fitting.

- The tube should then be rinsed wit acid solution then clean water.

- Dry the tube under gas flow.

- Reinstall the gas/liquid separator disc, pressing the tubing fully onto the barb fitting.

- The tube may the be replaced into the system.

- Perform a purge gas leak check as outlined later in this chapter.

IR Cell Maintenance

The infrared detector assembly is shown in Fig. 5.3. It is composed fo three distinct components. A light source contained in a beam chopper assembly, an aluminum block sample cell which is lined with a reflective gold foil and a detector cell which is wired to the detector electrometer. The gold foil and windows on the sample and detector cells are subject to contamination and gradual degradation due to corrosive by-products of persulfate oxidation. This is especially the case of applications involving sample with high chloride concentration (>2%) which produces chlorine gas when reacted with persulfate. For this reason, the cell should be

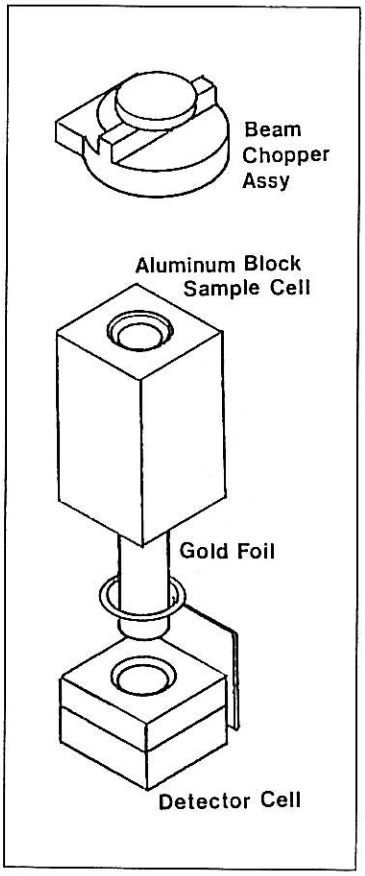


Fig. 5.3 Infrared Detector Assembly

inspected after every 2000 hours of operation. For applications where chloride is a very minor constituent in samples routinely analyzed, this inspection should be performed no longer than after 4000 hours of operation.

The maintenance should include cleaning of the cell windows with deionized water and acetone, and cleaning or replacement of the gold foil to assure a uniform, reflective surface. Later in this chapter, is a section entitled "Cleaning the Sample Cell of the Fuji ZFP-ZZ Infrared" and gives a detailed outline for gaining access to the IR detector.

IR Linearization Check

All non-dispersive infrared analyzers produce a non-linear response unless electronically corrected by a linearizer board or, in the case of the Model 700, the output response is corrected algebraically (best-fit, third-order polynominal).

If a linearity problem is suspected, contact the OI Service Department for assistance.

The detector in this instrument has been linearized over a range of 0-50 ug C and should remain linearized indefinitely. However, quality assurance practices and proper maintenance procedures should include routine linearity checks. The procedure that follows is recommended.

- Using a 1 milliliter sample volume (loop sampling or syringe is equivalent) calibrate the instrument on a 20-25 ppm C standard (TIC or TOC).

This is of course mid-scale (20-25 ug C) of the detectors range, so equivalent standards using appropriate sample volumes can be substituted.

- When calibration is completed by entering the scaling factor, run a 1-2 ppm C and a 50 ppm C standard to confirm linearity.

Non-Scheduled Maintenance
Mechanical

This section describes procedures for setting and testing certain mechanical components for proper operation if replaced during non-scheduled maintenance (troubleshooting).

Calibration of Acid Pump (#1)

Caution: Phosphoric acid is a corrosive substance, always wear appropriate chemical eye and skin protection when handling this material.

- Loosen the lock nut located on the threaded shaft at the bottom of pump.
- Remove the acid line from the top of the injection port cross-coupling and place the end in a large weighing boat.
- Confirm that the reagent bottle for acid is filled with reagent to be pumped.
- Press the **PRIME ACID** key until all the air bubbles are out of the pump lines, collecting dispensed acid in the weighing boat.
- Empty the weighing boat and dry thoroughly.
- Press the **PRIME ACID** key twice while capturing the effluent in the weighing boat. Using a 500 ul syringe, carefully measure all the contents in the weighing boat.

- If the volume is more than 200 ul, turn the union below the pump counterclockwise. If the volume is less than 200 ul, turn the adjustment nut clockwise.
- Repeat the above steps until the volume dispensed into the boat from two pulses is between 190 and 210 ul.
- When the pump dispenses the correct volume, tighten the locknut and check that correct volume is dispensed.
- Connect the acid line to the top of the injection port cross-coupling.

Calibration of Pump #2 (Persulfate)

- Loosen the locknut located on the threaded shaft at the bottom of pump.
- Remove the persulfate line from the digestion chamber cap and place the end in a large weighing boat.
- Confirm that the reagent bottle for oxidant is filled with reagent to be pumped.
- Press the **PRIME OXIDANT** key until all air bubbles are out of the pump lines collecting the dispensed oxidant in the weighing boat.
- Empty the weighing boat and dry thoroughly.
- Press the **PRIME OXIDANT** key twice while capturing the effluent in the weighing boat. Using a 500 ul syringe, carefully measure all the contents in the weighing board.

CAUTION: Sodium persulfate is a corrosive substance, always wear appropriate chemical eye and skin protection when handling this material.

- If the volume is more than 200 ul, turn the union below the pump counterclockwise. If the volume is less than 200 ul, turn the adjustment nut clockwise.
- Repeat the above steps until the volume dispensed into the boat from two pulses is between 190 and 210 ul.
- When the pump dispenses the correct volume, tighten the locknut and check that correct volume is dispensed.
- Connect the persulfate line to the top of the digestion chamber.

Purge Gas Leak Test

Refer to the appropriate plumbing schematics, in **Chapter 9**, for the system being checked.

- Perform a NORMAL POWER UP.
- Place instrument in MANUAL mode by pressing the **ANALYSIS MODE** key three times. Confirm that the red LED lamp on the front panel lights at the MANUAL position.
- Press key **#9** to turn on the purge gas.

- Fully open purge gas flow meter.

- Plug the sample drain line or the line from Valve B Port 2 and observe the PURGE rotometer. Flow should fall to 0 in approximately 1 minute. If it does not, the leak should be located with soap bubble solution and repaired.

- Press key #6 (SAMPLE DRAIN VALVE), and observe the rotometer. The float may come off the stop momentarily but should settle to 0. If it does not, there is a leak associated with the drain valve (Valve B). Locate and repair.

- Press key #6 again to return the valve to its original position.

This step is omitted if the instrument does not have the Process Sampling Capability.

- Press key #3 (SAMPLE LOOP VALVE), and observe the rotometer. The float may come off the stop momentarily but should settle to 0. If it does not, there is a leak associated with the sample loop valve (Valve C). Locate and repair.

- Press key #3 again to return the valve to its original position.

This step is omitted if the instrument does not have the Purgeable Organic Carbon Capability.

- Press key #2 (POC TRAP VALVE), and observe the rotometer. The float may come off the stop momentarily but should settle to 0. If it does not, there is a leak associated with the POC trap valve (Valve D). Locate and repair.

- Press key #2 again to return the valve to its original position.

- Press key #1 (PRIMARY TRAP VALVE), and observe the rotometer. The float may come off the stop momentarily but should settle to 0. If it does not, there is leak associated with the primary trap valve (Valve A). Locate and repair.

- Press key #1 again to return the valve to its original position.

A leak that is indicated by the flow meter but not detected or associated with valves or tube connections may indicate a leak between the inner and outer tubing of the permeation tube. Performing a carrier gas leak test will verify this.

- Remove the plug from the drain line and set the PURGE rate to 6 on the rotometer.

Carrier Gas Leak Test

Important: Always perform Purge Gas and Carrier Gas leak test SEPARATELY.

Refer to the appropriate plumbing schematics in chapter 9, for the plumbing being checked.

- Perform a NORMAL POWER UP.

- Place instrument in MANUAL mode by pressing the **ANALYSIS MODE** key #3 times. Confirm that the red LED lamp on the front panel lights at the MANUAL position.

- With an orange septum (OI Part #116-194) or appropriate device, plug off the gas flow from the vent line of the permeation tube.

If you unit has Purgeable Organic Carbon capability, set the furnace temperature to 0 and place a 5 mm septum (same as the injection port septum) in the side arm of the

oxygen inlet tee (the arm with the 1/16 inch line) to plug the oxygen line. Remember to remove the septum from the tee and set the furnace temperature back to 800°C when test is complete.

- Observe that the detector flow drops to zero. If it does not, the leak should be located with soap bubble solution and repaired.

- Press keys **#1** and **#2** and observe the detector flow. The float may come off the stop, but should settle to 0. If it does not, there is a leak associated with the primary trap valve (key #1) or the POC trap valve (key #2). Locate and repair.

A leak that is indicated by the flow meter but not detected or associated with the Primary trap valve, POC trap valve or IR detector connections may indicate a leak between the inner and outer tubes of the permeation tube. Performing a purge gas leak test will verify this.

- Press keys **#1** and **#2** again to return the trap valves to their original positions.

- Remove the septum from the vent line.

- Press key **#1** and set the DETECTOR flow rate to 5 on rotometer.

Actuator Gas Leak Test

- Adjust both knobs on two-tube flowmeter on Model 700 front panel to maximum opening (fully counterclockwise).

- Place instrument in MANUAL mode by pressing the **ANALYSIS MODE** key 3 times. Confirm that the red LED lamp on front panel lights at MANUAL position.

- Plumb a test flowmeter between the actuator gas source and the actuator gas inlet.

- Confirm that the float on test flowmeter falls to 0. If it does not, find any leaks in actuator plumbing using a soap bubble solution. Tighten, repair, or replace components as necessary to remove leaks.

- Confirm proper rotation of 6-port valves. Activate each valve by pressing the following keys while watching for proper rotation and leave activated.

Key	Valve	Rotation	Display
1	A (Prim Trap)	Counter-clockwise to activate	TV1>
6	B (Drain)	Rotates clockwise to activate	DV>
3	C (Sample Loop)	Rotates clockwise to activate	SV>
2	D (POC Trap)	Rotates counter-clockwise to activate	TV2>

Rotate all these valves to activated position if they were not left in that position from the previous step (confirm TV1>, DV>, SV>, TV2> on screen), and confirm that float on test-flowmeter slowly falls to zero. If it doesn't, then look for leaks in actuator plumbing using a soap bubble solution. Tighten, repair, or replace components as necessary to remove leaks.

Calibration of Sample Loops (0.3 ml - 10 ml)

Place an empty weighing boat on the weighing pan of the balance and record the tare weight.

- Place instrument in MANUAL mode by pressing the **ANALYSIS MODE** key 3 times. Confirm that the red LED lamp on front panel light at Manual position.
- Press the **#6** key to activate the sample drain valve (B).
- Press the **#9** key to activate the sample purge valve.
- Press the **#1** key to activate the primary trap valve (A).
- Disconnect Valve C Port 1 from Valve B Port 3.
- Install the sample loop to be calibrated between Valve C Ports 3 and 6.
- Place the loop sampling tube into a container of reagent water, and press the **SAMPLE PUMP ON/OFF** key to load the sample loop. Allow the pump to run until the loop has filled. Complete filling can be visually verified.
- Place the tube from Valve C Port 1 into the weighing boat and press key **#3** to activate the sample loop valve (C). Water from the sample loop will flow into the weighing boat.
- Once the water has been collected, press key **#3** to return Valve C to its original position.
- Place the weighing boat on the balance pan, and record the weight.
- Dry the weighing boat and repeat the above steps until satisfactory replication is obtained.
- Average the measured masses of water. The average mass in grams is considered equal to the loop volume in milliliters (1 g = 1 ml of water).
- Reconnect Valve C Port 1 to Valve B Port 3.

Calibration of Microliter Sample Loops (40 ul and 100 ul)

- Install loop to be calbrated directly onto Sample Valve Ports 3 and 6. Check for leaks by placing sipper tube in a container of water and pressing the **SAMPLE PUMP ON/OFF** key. If bubbles are seen, hand tighten fittings until bubbles stop.
- Review SET SYSTEM CONFIGURATION menu. Enable SAMPLE PUMP, SAMPLE LOOP VALVE and ANALYZE TIC ONLY.

- Set sample volume to 1.00 millileter.
- Cycle the instrument 3 times with a 500 ppm C solution of sodium carbonate.
- Average the millivolt responses.
- Prepare the instrument for syringe injection by reviewing SET SYSTEM CONFIGURATION menu. Disable SAMPLE PUMP, SAMPLE LOOP VALVE, and AUTO-RUN.
- Using a 100 ul syringe, inject the equivalent volume (i.e. 40 ul if calibrating the 40 ul loop, or 100 ul if calibrating the 100 ul loop) of the 500 ppm C sodium carbonate standard. Repeat injection and average the millivolt responses.
- Solve for the sample volume (in microliters) using the following formula.

$$\text{Sample Loop Volume} = \frac{C \times A}{B}$$

Where A = Average millivolt response of loop
B = Average millivolt response of syringe injection
C = Syringe volume, in **microliters**

- Label the loop with the calculated volume.

The final concentration, as calculated by the microprocessor, is the micrograms of carbon detected divided by the volume of the sample entered with the **SET SAMPLE VOLUME** key. Thus, the volume of the microliter loop as calculated in the previous step must be divided by 1000 to obtain the proper volume dimension (i.e. milliliters) since 1 ppm C = 1 ug C/ml and the microprocessor expects a volume in milliliters when making the final concentration calculation.

- Repeat procedure for the remaining loop, substituting the correct volume as needed.

Molecular Sieve Trap Cleaning

In the unlikely event that reagents or a saline sample gets pushed through the molecular sieve trap, the trap should be washed with water and dried before heating it to desorption temperature. In this case perform the following steps:

- While power is on, set the PRIMARY TRAP TEMP to 0.
- Confirm that the instrument is in the TIC/TOC Analysis Mode and press CLEAR.
- Turn off the power to the instrument and leave gas pressure on.
- Disconnect the fittings at Valve A Port 4 and Valve A Port 5.
- Using a syringe and elastic tubing for connection, force clean water through Valve A Port 4 until several milliliters drip out of Valve A Port 5.
- Pull the syringe plunger to pull air in a reverse direction (into Port 5) through the trap.

- Reconnect Valve A Port 4 to its original gas line from the carrier gas flowmeter.
- Allow carrier gas to flow through the trap in this manner until droplets cease to form at the open Valve A Port 5.
- Perform a #1 KEY POWER UP.
- Set the PRIMARY TRAP TEMPERATURE to 90° C and press the **CLEAR** key.
- Wait several minutes until the carrier gas carries any additional water out through Valve A Port 5.
- Repeat this heating procedure for a few progressively higher temperature settings (for example: 110°, 140°, 170°, 200°) until 200° C is set as the trap temperature.
- Reconnect Valve A Port 5 with the original tube connector.

Flow Rates

Purge Gas

- Press the **SELECT ANALYSIS MODE** key until the instrument is in the MANUAL MODE.
- Press key **#9**.
- Set the Purge flow rate to 6 on the Purge rotometer.

Primary Trap

- Press the **SELECT ANALYSIS MODE** key until the instrument is in the MANUAL MODE.
- Press key **#1** (this places the Primary Trap in-line with the detector flow) and set the Detector rotometer to 5.
- Press key **#4** (this heats the Primary Trap to 200° C) and observe the rotometer, it should not drop below 4.5 on the rotometer.
- Press keys **#4** and **#1** again. The ball should not rise above 6 on the rotometer.
- Any variationbelow the minimum flow or above the maximum, flow necessitates changing the Primary Trap (OI Part #167-149).

POC Trap

With the Primary Trap flow set as described above, perform the following:

- Press key **#2** (this places the POX trap in-line with the detector flow) and observe the rotometer, it should not drop below 4.5.
- Press key **#5** (this heats the POC trap to 180° C) and observe the rotometer, it should not drop below 4.
- A flow of less than 4, indicates the need for replacing the POC Trap.

- Press the **SELECT ANALYSIS MODE** key for the desired analysis mode.

Cleaning the Sample Cell of the Fuji ZFP-ZZ Infrared (IR) Detector

Important: Please read these instructions through once before attempting to perform this procedure. Note orientation of components before disassembly. (Refer to Fig. 5.4)

- Turn off and unplug the instrument.
- Disconnect the pneumatic and electrical connection from the rear of the IR, noting their placement.
- Unscrew and remove the 4 screws that hold the IR enclosure to the Model 700 chassis. Remove the enclosure from the Model 700.
- Remove the 8 screws from each end plate of the enclosure and carefully lay each plate down.
- Unscrew the 4 nuts on the studs that hold the IR chassis to the enclosure. Remove the chassis from the enclosure allowing one of the enclosure end plates to follow the IR chassis out.

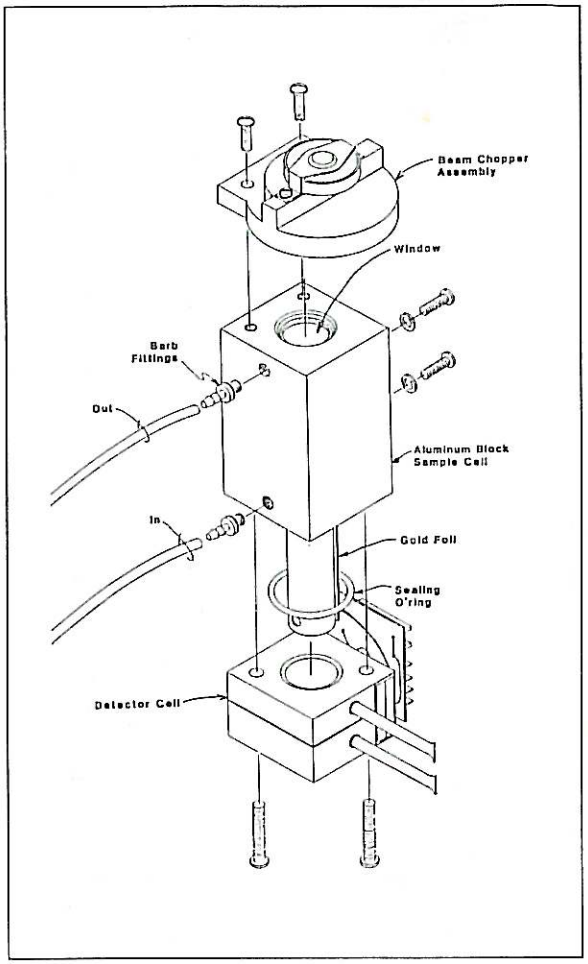


Fig. 5.4 Infrared Detector Assembly

- Locate the IR sample cell (the rectangular aluminum block with two white tubes connected). The chopper motor is located on the top and the detector on the bottom.
- Label the two white tubes (OI Part #111-401) in and out.
- Unscrew the two screws holding the chopper motor to the top of the sample cell (OI Part #172-833) and carefully set the motor aside.
- Unscrew the two screws holding the sample cell to the IR chassis and carefully turn the cell and unscrew the two screws holding the detector to the cell.
- Carefully remove the gold foil (OI Part #173-279) from inside the sample cell. Rinse foil off with clean water and dry.
- Look through the cell window (OI Part #173-287) and insure the window is clear. If the window is fogged the cell will need to be replaced.

- Rinse the interior of the sample cell with clean water followed by an acetone or methanol rinse to quickly dry out the water. Be careful to dry the window **WITHOUT** scratching it.

- Reinstall the gold foil in the sample cell, making sure the holes in the foil line up with the holes in the cell.

- Attach the detector to the bottom of the sample cell (the end without the window) making sure the O-ring (OI Part #116-400) is in place. The detector should be screwed down evenly to form a leak-free seal.

- Attach the sample cell and detector to the IR chassis, then attach the chopper motor to the top of the cell assembly.

- Connect the two white tubes to the barbed fittings on the sample cell with the carrier IN tube closest to the detector cell.

- Being sure that the IR power leads do not short out against one another or against the Model 700 chassis, plug in and power up the Model 700.

- Reconnect the carrier IN and carrier OUT tubes on the rear panel of the IR and perform the carrier gas leak test as outlined earlier in this chapter.

- Turn power off and unplug the Model 700 and disconnect the carrier gas tubes from the IR rear panel.

IR baseline will drop as IR case is purged of ambient CO_2. After IR has equilibrated, baseline zero may need to be adjusted to 5-10 mV.

- Install the IR back into its enclosure by first pushing one of the end panels through the box.

- Replace the chassis on the studs and attach the four lockwashers and nuts.

- Reattach the end panels to the IR enclosure and install in the Model 700. Connect the pneumatic and electrical connections.

- Turn on the Model 700 and adjust the IR output to about 60 mV.

Non-Scheduled Maintenance Electronic

This section describes procedures for testing and calibration of electronic components for proper operation if replaced during non-scheduled maintenance (trouble-shooting).

Maintenance requirements of the instrument are not extensive. Visual inspections are comprised mostly of the suggested maintenance procedures. Electronic re-alignment is not generally required on a regular schedule and need not be performed unless certain circuit boards or components are replaced or the unit shows signs of misalignment in the analog circuitry.

Visual Inspections

- Inspect the condition of the thermocouple junction at each of the heated devices. Check for broken junctions or incorrect positioning of the thermocouples in the digestion block heater and optional POC furnace. Also check the mechanical

integrity of the trap mounted thermocouples. Check and tighten where necessary the terminal block screws on the thermocouple MUX board at the points where thermocouple wires attach.

- Inspect the socketed integrated circuit chips (ICs) on the processor board and the I/O board and gently press down or re-seat any of them that appear to be working loose. It is a normal occurance for ICs to gradually work loose in sockets due to PC board thermal expansion effects over long-term.

- Visually check all ribbon cable and round cable connectors to see that they are fully plugged into their mating connectors.

Electronic Alignment Procedure

The following alignment procedure is provided so that a field alignment of the electronic control system may be performed. It is recommended, however, that this only be performed by a factory-trained electronic technician. In addition, electronic alignment of the control electronics is not normally required on a regular basis to maintain accuracy of results. The IR analyzer may require periodic alignment should the internal linearizer circuit response drift over long-term operation.

The alignment procedure consists of a general operational checkout sequence to verify that the system responds properly and that the different system functions are operational. An alignment of the analog circuitry on the I/O board is followed by an adjustment procedure for the overtemperature shutdown circuit on the thermocouple MUX board. Refer to the PC board schematics and parts locations in **Chapter 9**.

Equipment required: Digital Multimeter, Oscilloscope, DC Millivolt Calibration Source, Jumper Clips and Parts Location Drawings.

The procedure is as follows:

- Turn off the main power switch and unplug the power cord.

- Unplug the ribbon cable on the I/O board leading to the AC power control board to prevent any heater operation during this phase of the checkout. Also temporarily disconnect the 50 conductor ribbon cable between the processor board and the I/O board.

- Position the NORMAL/TEST switch located on the processor board, under the main power switch, to the TEST position.

Note: This switch MUST be set back to Normal when the AC power control board ribbon cable is re-attached to the I/O board. If the unit is run in TEST with the heaters operating, the trap transformers will soon overheat due to lack of processor synchronization with the line frequency.

- Reconnect the line cord and turn the main power switch back on.

Electronic Alignment Checkout

- Reconnect line load and turn on power to the system. Verify that the following functions are operational:

  - +5 volt LED on processor board is on.

- +5 volt, +12 volt and +15 volt LEDs on I/O board are on

- all 8 AC power control indicator LEDs on I/O board are on

- 4 green DC power control LEDs on the middle row of the I/O board are on

- the top 2 red LEDs and the bottom two green LEDs on the display board are on

- the alpha-numeric LED display shows the proper message and time 00:00:00

· Check that the lower right display character toggles position when the **RUN/STOP** key is pressed.

· Check that every red LED in the right column of LEDs on the display board (Analysis Mode LEDs) can be lit using the **SELECT ANALYSIS MODE** key on the keypad.

· Verify that every red LED in the left column of LEDS (Display Model LEDs) can be lit using the **SELECT DISPLAY MODE** key on the keypad.

Processor Board To I/O Board Communication Check

· Turn off power. Connect the ribbon cable from the processor board to the I/O board. Turn power back on.

· Briefly verify that all functions described in the Electronic Alignment Procedures are the same except for the red AC power control LEDs on the I/O board. Verify visually that the red LED closest to the front of the I/O board is flashing about 4 Hz, 50% duty cycle (Heartbeat).

+12 Volt DC Output Check

· Select MANUAL mode using the **SELECT ANALYSIS MODE** key. Verify that bottom 2 red LEDs on display board are lit.

· Press the following keys and verify that the corresponding green LEDs turn On/Off. Refer to Fig. 2.9 for LED functions. Also check the display to determine that the proper control state is indicated.

Key **#1** - Primary Trap Valve - TV1
Key **#2** - POC Trap Valve - TV2
Key **#3** - Sample Loop Valve - SV
Key **#6** - Sample Drain Valve - DV
Key **#7** - Acid Injection (< 1/2 sec pulse)
Key **#8** - Oxidant Inject (< 1/2 sec pulse)
Key **#9** - Sample Purge Valve - PV

+120 Volt Pump Control Check

· Refer to **Chapter 2**, for location of the red AC power control indicators and their functions. Press the **SAMPLE PUMP ON/OFF** key and verify that the pump LED goes On/Off.

Event Timer Function

- Press the **SET SYSTEM CONFIGURATION** key and set the following:

  Enable sample pump
  Enable sample loop valve
  Enable Auto-Run
  Disable Auto-Print. Verify that green AUTO-PRINT LED goes off
  Enable autosampler
  Enable Ready/Standy status over-ride
  Disable TIC only
  Disable TOC only
  Disable ampule analysis

- Select normal TIC/TOC analysis mode (top right LED on). The unit should now run through a complete analysis sequence and repeat when the **RUN/STOP** key is pressed. Try it. The red RUN/STOP LED should begin flashing at 1 Hz.

Line Synchronization Circuits Check

- Turn on the power to the unit and reconnect the AC power control board ribbon cable to the I/O board. Set the TEST/NORMAL switch on the processor board back to the NORMAL position.

- Turn the power back on. Verify that the red LED for the spare AC power control is flashing which is an indication of proper line frequency synchronization signals to the processor.

Temperature Control Circuit

- Select the Manual mode of operation with the **SELECT ANALYSIS MODE** key. Using the **SELECT NEXT DISPLAY** key, go to the heater 1, heater 2 temperature display. If the fan is on, the heater 1 temperature should read a few degrees above ambient.

- Heat the primary trap using the **PRIMARY TRAP HEAT/COOL** key while watching the trap temperature display. Verify that the trap heats up to 200° in approximately 15 to 20 seconds. Cool the trap and verify that the fan comes on and the trap cools to a few degrees above ambient in about 30 seconds.

- Select the next display to view the heater 3 and heater 4 temperatures, the digestion block temperature and the POC furnace (optional) respectively. The temperature of heater 3 should be climbing to 100° C and should eventually stabilize at that temperature (about 10 minutes).

- Select the normal TIC/TOC ANALYSIS mode.

Keypad Functions

- Confirm that the timer is stopped. Press the following keys and check for the proper response or display message.

- Press the **10X SENSITIVITY** key and verify that the green 10X SENSITIVITY LED toggles ON/OFF.

- Press the **SELECT ANALYSIS MODE** key and select Manual mode.
- Verify that pressing the **SELECT NEXT DISPLAY** key changes the display messages.
- Re-select Normal TIC/TOC analysis mode with the **SELECT ANALYSIS MODE** key.
- Press the **RUN/STOP** key to start clock. Press the **RAPID CLOCK ADVANCE** key and verify that the timer counts 5 x normal speed.
- Press the **PRIME ACID** key and verify that the green acid inject LED flashes.
- Press the **PRIME OXIDANT** key and verify that the green oxidant inject LED flashes.
- Verify that the **SET ACID VOLUME** key functions.
- Verify that the **SET OXIDANT VOLUME** key functions.
- Verify that the **SET SAMPLE VOLUME** key functions.
- Verify that the **SET TIMES** key functions.
- Verify that the **SET TEMPERATURE** key functions.
- Press the **SET SAMPLE VOLUME** key. Check each of the digit keys **0** to **9**, the **CLEAR ENTRY** key, the **.** (decimal) key and the **ENTER** keys to see that they all function properly.
- Start the timer with the **RUN/STOP** key. Press the **CLEAR** key and verify that the timer resets to 0 and stops counting.

I/O Board Field Alignment Procedure

- With DVM, adjust R109 for 2.50 volts between U27(6) and ground test point.
- Adjust R114 to 2024 mV between test point 5 and test point 6 (ground).
- Adjust R111 for 520 mV between test point 7 and test point 6 (ground).
- Short together pins 1, 2 and 3 of J9.
- With 10X sensitivity ON, adjust R91 for 0.000 volts between R86 and R87 measured at the resistor ends furthest from U23 (right end). Alternately, select IR mV display and adjust for 000 mV.
- Short together J9 pins 2 and 3 (-input, ground). Connect MVS ground to J9 2 and 3, MVS output to J9 pin 1 (+input). Select IR mV display mode and set 10X sensitivity ON. Adjust MVS for 100.000 mV as seen on display. Turn 10X sensitivity OFF and adjust R93 for 100.000 mV on display.

- Short together J9 pins 1 and 3 (+input, ground) and connect to MVS ground. Connect MVS output to J9 pin 2 (-input). Select IR mV display mode and 10X sensitivity ON. Adjust MVS for -100.000 mV on display. Turn 10X sensitivity OFF and adjust R101 for 100.000 mV on display.

- Connect MVS ground to J9 pin 3. Connect another wire from MVS ground or (-) output to J9 pin 2. Connect MVS (+) output to J9 pin 1. Adjust MVS for 100.0 mV output measured between J9 pins 1 & 2. Turn 10X sensitivity ON from keypad. Select IR mV display and re-adjust R111 for 100.00 mV on display.

- Unplug the ribbon cable from the thermocouple MUX board. Connect the MVS ground lead to the ground test point on the I/O board. Connect MVS (-) output to the right end of R3 and the MVS (+) output to the right end of R4. Adjust MVS for 1000.0 mV output. Select diagnostics display, ADC2 DATA, and re-adjust R114 for 1000 on the display.

Zero Adjustment:

- Select the ambient temperature display and note the readings (23-30° C usually).

- Select the heater 3, heater 4 temperature display.

A solid jumper wire must be installed between terminals 7 and 8, the POC furnace thermocouple location, on the thermocouple MUX board. If the POC option is not installed, there should already be a jumper here.

- Adjust the zero control potentiometer R33 until the heater 4 temperature matches the ambient temperature -0 + 1 degree.

- Re-check the ambient temperature reading.

Non-Scheduled Maintenance Options

POC Catalyst Tube

The POC catalyst tube (OI Part #168-189) for the most part, is maintenance free. It is important to remember however, that the cobalt, iron, nickel catalyst must be exposed to an oxidizing environment, otherwise, POC as well as the TOC and TIC samples passing through the tube can be negatively affected. Thus, the POC furnace must operate at 800-900° C and oxygen, at 10 psi delivery pressure, must be supplied to the furnace via the auxiliary gas inlet to assure optimum oxidation conditions.

Should the catalyst be suspected as the cause of low POC recoveries, the catalyst tube is changed as follows:

- Press the **SET TEMPS** key and advance to the POC FURNACE TEMP display.

- Turn off the furnace by entering 0° C from the keypad and allow furnace to cool to ambient temperature.

- Loosen (but do not remove) the 1/4" (large) Swagelok nuts at the unions on each end of the catalyst tube.

The internal temperature of the furnace may be monitored in the DIAGNOSTICS INFORMATION display mode as HTR4.

- Remove the union on the inlet of the tube (towards front of instrument).
- The catalyst tube should now slide out the front of the furnace.
- If tube is to be re-packed use catalyst (OI Part #154-740) and quartz wool (OI Part #144-501) to retain catalyst inside of the tube. Installation of new catalyst tube is the reverse of these steps.

CAUTION: Tube may still be hot, handle with care.

- Perform detector gas leak check as outlined earlier in this chapter.

Ampule Breaking Assembly

When the Model 700 is configured to analyze ampules as described in **Chapter 3**, the system should be free of leaks when an ampule or the standarization vial is mounted in the breaking assembly. This can be verified by performing purge gas leak check as described earlier in this chapter.

It is also necessary to inspect the latex O-rings (OI Part #116-350) in the cutter plunger barrel on occasion for cracks or splits which would allow sample gas to be lost. These O-rings as well as the O-ring (OI Part #116-186) used to seal the cutter plunger barrel to the ampule collar should be very lighty coated with silicone stopcock grease to facilitate the sliding of the cutter plunger barrel.

Printer Checkout

The printer supplied with the printer option (OI Part #169-004) has a programmed self test routine. It is activated as follows:

WARNING: Performing a #0 key power up will reset all operating parameters to default values

- Plug the printer power cord into the auxiliary power receptical located on the right, rear panel of the Model 700, and perform a #0 key power up. Do not connect printer interface cable to the instrument at this time.
- While pressing the **LINE FEED** button on the printer, turn the printer power switch ON.
- Printer should perform a continuous series of line feed/print operations, an example of which is given in the printer operations manual. If the printer does not execute the self test as specified in the printer operations manual, a problem exists within the printer.
- Turn the printer power switch OFF to stop printer self test.
- Connect the printer to the Model 700, with the interface cable, to the PRINTER output connector on the rear panel of the instrument.
- Press printer power switch to ON and confirm that printer is on-line as indicated by the light on printer display panel.

LAST ANALYSIS RESULTS time (HH:MM:SS) will be time elapsed since power up of instrument to PRINT test.

- Press the PRINT switch on the display board of the Model 700 and verify that printer prints the analysis conditions shown in Fig.5.5. Perform this test 3 to 4 times.
- If a fault is indicated in this print test, then printer cable or I/O board should be suspected as problem.

```
                    *** MODEL 700 TOC ANALYZER ***

CALIBRATION FACTORS:

IC BLANK = 000000 mV   OC BLANK = 000000 mV  I.R. OFFSET = 000000 mV
STD. MASS = 000000 mV STD. AVG. = 1.00000 mV  SCALING FACTOR = 0.05000 ug/mV

VOLUMES:

SPL. VOL. = 1.00000 ml   ACID VOL. = 00002 X 100 ul          OXID. VOL. = 00010 X 100 ul

LOW/HIGH ALARM SETPOINTS (ppm C):

TIC LO = 000000      TIC HI = 000000      TOC LO = 000000      TOC HI = 000000

TIME PRESETS:

EXTD. REACTION TIME 00:00:00 EXTD. PURGE TIME 00:00:00


LAST ANALYSIS RESULTS:

SPL#  00001      00:00:41      TIC = 000000 mV      000000 ug C      000000 ppm
SPL#  00001      00:00:43      TOC = 000000 mV      000000 ug C      000000 ppm
```

Fig. 5.5 Sample Analysis Printout

Autosampler

- The autosampler stage optical sensor and shuttles should routinely be cleaned of any sample spillage with a damp, squeeze dried sponge, then wiped with a dry cloth.

- See the supplemental Autosamapler Instruction Manual for further maintenance and repair procedures.

Chapter 6
Troubleshooting

The following is a list of instrument symptoms, and their most probable causes and corrections. Before using this section, the operator should become thoroughly familiar with the operation and maintenance information contained in previous chapters.

System Performance Symptoms

SYMPTOM	PROBABLE CAUSE	CORRECTIVE ACTION
No response	No Carrier and Purge gas flow	Check flowmeter settings. See Chapter 5
	Wrong reagents being used	See Chapter 1
	No actuator gas	See Troubleshooting for specific components later in this chapter
	Defective Trap	Replace Trap
	Actuator for Valve A not rotating	See Troubleshooting for specific components later in this chapter
	Valve clogged	Review 6-port valve maintenance in Chapter 5
	Leak in sample pneumatic lines or valves	Perform leak checks outlines in Chapter 5 service valves as necessary
	IR cell is contaminated	See Cleaning Procedure for Fuji ZFP-ZZZ IR in Chapter 5
	Trap does not heat	Follow check procedure in Chapter 5 for Temperature Control Circuit to determine if heating power is reaching trap
	Trap heats continually	Faulty PC board. Perform Temperature Control Circuit Checkout in Chapter 5 to determine exact solution
Non-reproducible response for both TIC and TOC	Sample volume not constant	Improve syringe technique

SYMPTOM	PROBABLE CAUSE	CORRECTIVE ACTION
		Check sample loop for complete filling when sample pump is on
		Inspect pump cartridge for leaks or wear (see Sample Pump Housing Maintenance in Chapter 5)
		Leak-check sampling tubing from loop injection port to sample pump (refer to Fig. 3.4)
	Improper flow rates	Check flow rates and make adjustments as outlined in Chapter 5, Trap Flow Rates
	Leak in system	Perform Purge gas and Carrier Gas Leak checks (Chapter 5). Finger-tight connections, valves and O-ring seals are suspect
	Valve leaking	Tighten cap-screws in top of valve. See 6-Port Valve Maintenance in Chapter 5
	Insufficient acid to completely liberate CO_2	Increase acid volume using SET ACID VOL key. Check acid volume following procedure in Chapter 5
	Insufficient purge time for high TIC samples	Extend purge time*
	Insufficient reaction time for complete TOC oxidation	Extend reaction time*
	Oxidant from previous sample not completely drained	Reposition tube inside digestion vessel (see Digestion Vessel Maintenance given in Chapter 5)
	Contaminated Digestion Vessel	Allow instrument to perform Clean Water Cycling as described in Chapter 4
		If gross contamination is present, disassemble digestion and clean with 400 grit sand paper and rinse with hot water. Reassemble according to the procedure

**See Confirming Recoveries in Difficult Samples (Chapter 4), to optimize extended purge or reaction times.*

SYMPTOM	PROBABLE CAUSE	CORRECTIVE ACTION
		in Chapter 5 (Digestion Vessel Mainenance) then perform Clean Water Cycling as described in Chapter 4)
Non-linear response for TIC and TOC	IR baseline set too high	Adjust IR zero to be 5 - 10 mV in Ready condition (see Chapter 5)
	Blanks not properly determined or entered	Determine and enter blanks
	Carbon mass exceeds linear range of detector	Refer to tables in Chapter 4 for selection of sample volume. Reduce sample size or dilute sample
	IR not properly linearized	Confirm linearity following IR Linerization (see Chapter 5)
Negative values display or printed	Improper blank mV value in memory (too high)	Determine and enter proper blank value
Offscale response when trap heats with no sample	Water in trap (reagent and sample volumes too large)	Reduce total volume to less than 16ml. Clean trap according to procedure outline in Chapter 5, Molecular Sieve Trap
	Gross purge gas contamination	Install in-line ascarite scrubber or use higher quality gas
	Carbon scrubber loaded	See Activated Carbon Scrubber Maintenance in Chapter 5
	IR baseline too high	Reset IR zero as described in Chapter 5
	IR cell is contaminated	Service IR cell
Low response for both TIC and TOC	Improper flow rates	Adjust flow as outlined in Chapter 5
	Trap(s) are to restrictive	Clean or replace
	Erroneously high blank values entered for TIC and TOC	Determine and enter proper blanks (see Chapter 4)

SYMPTOM	PROBABLE CAUSE	CORRECTIVE ACTION
	Erroneously low calibration constant entered	Determine and enter proper calibration constant. Refer to Chapter 4 for Calibration procedure
	Bad molecular sieve trap	Replace
	Erroneously high sample volume entered	Enter proper sample volume (see Chapter 2)
Low response for TIC with normal or reproducible response for TOC	Purge gas or Carrier gas leak	Perform Purge Gas and Carrier Gas leak tests (Chapter 5)
	Restriction in sample gas lines	Clean valves and pneumtic lines. See Chapter 4, 6-Port Valve Maintenance
	Insufficient acid addition to completely librate CO_2	Increase acid volume
	Insufficient purge time	Extend purge time
	Improper acid reagent	Confirm that 5% (vol/vol) phosphoric acid is being used
	Faulty acid pump	Check acid pump calibration (see Chapter 5)
	Faulty acid pump circuit	Follow procedure for +12 VDC output Check and Keypad Function Check in Chapter 5 to determine if 12 VDC is reaching pump
	TIC mass exceeds linear range detector	Refer to tables in Chapter 4 for selection of sample size. Reduce sample size or dilute sample
Low response for TIC with high response for TOC	Insufficient acid to completely liberate CO_2	Increase acid volume
	Insufficient purge time for high TIC samples	Extend purge time
	Faulty acid pump	Check acid pump calibration (Chapter 5)
Low response for TOC with normal response for TIC	Incomplete oxidation, not enough oxidant	Increase oxidant volume

SYMPTOM	PROBABLE CAUSE	CORRECTIVE ACTION
	No persulfate reagent or improper reagent in oxidant reagent bottle	Confirm that correct persulfate solution is in oxidant reagent bottle. See Chapter 1 for reagent and materials required
	Incomplete oxidation, insufficient reaction time	Extend time of reaction
	Faulty oxidant pump	Check oxidant pump calibration. See Calibration of Pump #2 in Chapter 5
	Faulty oxidant circuit	Follow procedure for +12 VDC Output Check and Keypad Function Check in Chapter 5 to determine if 12 VDC is reaching pump
	TOC mass exceeds linear range of detector	Refer to tables in Chapter 4 for selection of sample size. Reduce sample size or dilute sample
	Digestion vessel not heating	Follow procedure in Chapter 5, Temperature Control Circuit, to determine if power is reaching digeston vessel heater block
	Purge valve does not shut off completely during digestion (i.e. ball on rotometer off of stop)	Replace purge valve if float on purge flowmeter does not stay at zero during sample digestion
Low TOC response with high TIC response	Oxidant from previous sample not completely drained	Reposition tube inside digestion vessel. See Digestion Vessel Maintenance in Chapter 5
	Reagent bottles switched	Check Purge gas flow during draining, note any excessive restriction preventing good draining of vessel. Confirm acid and persulfate in correct bottle to respective pumps
High response for TIC and TOC	Erroneously low blank values entered for TIC and TOC	Determine and enter proper blanks
	Erroneously high calibration constant entered	Determine and enter proper calibration constant

SYMPTOM	PROBABLE CAUSE	CORRECTIVE ACTION
	Erroneously low sample volume entered	Enter proper sample volume
	Flow rate too low for detector carrier gas	Adjust according to procedure in Chapter 5
	System contamination	Perform visual inspection of all surfaces which contact sample and clean as needed with hot water. Perform clean water cycling routine described in Chapter 4
High TIC blanks	CO_2 in acid	Confirm reagent bottle purging. Purge CO_2 from acid
	CO_2 in purge gas	Install ascarite scrubber in-line or use higher quality gas
	Sample loop enabled and injecting room air	Disable sample loop while running blanks
High TOC blanks	CO_2 in oxidant	Confirm oxidant bottle is being purged. Purge CO_2 from reagent
	CO_2 in purge gas	Install ascarite scrubber in-line or use higher quality gas
	Organic carbon in oxidant	Clean oxidant organics. See Reagents and Materials section in Chapter 1
	Organic carbon in acid	Clean acid of organics. See Reagent and Materials section in Chapter 1
	Digestion vessel contaminated	Cycle analysis mode with extended digestion time. See Clean Water Cycling in Chapter 4 to clean vessel or remove vessel and clean with 400 grit sand paper then rinse with hot water
	Drain line in digestion vessel not positioned properly (i.e. carryover)	See digestion Vessel Maintenance in Chapter 5
	Organic carbon breakthrough from activated carbon scrubber	Bake out or replace scrubber (Chapter 5, Activated Carbon Scrubber Maintenance)

SYMPTOM	PROBABLE CAUSE	CORRECTIVE ACTION
Erroneous values for TC only analysis	When system is in TC mode, all possible TIC/ TOC blank problems must be considered	When in TC mode, test solutions for high TIC blanks as well as for high TOC blanks
Low POC response	Erroneously High blank values entered for POC	Determine and enter proper blanks
	Furnace not at 800° C - 900°C	Check furnace temperature setting and set to correct temperature if too low
		Check thermocouple for closed circuit and proper connection to MUX board. Replace thermocouple if faulty
	Poisoned POC catalyst	Replace catalyst tube with new catalyst (see Chapter 5)
	Oxygen reaction gas off or improper flow	Confirm 10 psig regulator pressure and ca. 75 ml/min O_2 flow at oxygen tee fitting
	Flow rates to low	Adjust flows as outlined in Chapter 5. Visually inspect pneumatic lines for restrictions
	Insufficient purge time	Extend purge time
High POC response	Gas leaks	Perform Purge Gas and Carrier Gas leak checks outlined in Chapter 5. Take corrective actions as necessary to fix leaks
	Erroneously low blank values set for POC	Determine and enter proper blanks
	Improper flow rates	Adjust flow rates as outlined in Chapter 5
	Hydrocarbon contamination in purge gas	Bakeout hydrocarbon scrubber. See Activated Carbon Scrubber Maintenance, Chapter 5
		Add an in-line gas scrubber
Instrument will not power up	Unit not plugged in to appropriate line voltage	Check power cord connection
		Check power breaker to plug outlet. Reset if tripped

	Blown fuse	Check A/C power control board fuses and replace if blown. See Chapter 2 for location
	No gas pressure (less than 15 psig) to instrument/ pressure cut off switch	Turn on gas pressure to instrument and set regulator to 30 psig

Specific Component Symptoms

SYMPTOM	PROBABLE CAUSE	CORRECTIVE ACTION
6-Port Valve	No actuator gas	Check actuator gas pressure (i.e. 30 psig) and flow at bulkhead
	Faulty actuator	Manually test rotation by connecting actuator gas directly to one gas inlet on actuator, then the other. Rotation should occur as pressure is applied to each inlet while letting the other exhaust. Also see Actuator Gas Leak Test in Chapter 5
	Faulty solenoid	Follow procedure in Chapter 5, Keypad Fuctions, to determine if 12 VDC is reaching solenoid
	Faulty PC board	Follow procedure in Chapter 5, +12 VDC Output Check, to determine if 12 VDC is reaching solenoid
Sample does not properly aspirate into instrument from sample bottle or autosampler	Incorrect sample pump time/sample volume entered	Enter correct values
	Worn pump cartridge	Replace pump cartridge as described in Chapter 4, Sample Pump Housing Maintenance
	Leak in sampling line	Leak-check sampling tubing from loop injection port to sample pump (refer to Fig. 3.4)
	Sample loop not properly tightened	Check sample loop connections for finger-tightness
	Pump head tubing pinched shut from lack of use	Service or replace pump tubing. See Chapter 5

SYMPTOM	PROBABLE CAUSE	CORRECTIVE ACTION
	Sample pump does not rotate	Follow procedure in Chapter 5, +120 volt Pump Control, to determine if power is reaching the pump
	For autosampler using septum piercing option, fill gas may be needed	Supply a separate N_2 or He gas supply (approx. 5 mlmin) to TEE fitting on septum piercing assembly
Peaks have shoulder-like tails	Excess water vapor is passing into detector	Change permeation tube See Chapter 4, Gas Permeation Tube Maintenance
		Do not heat digestion vessel to over 100° with sample volumes greater than 2 ml
	Primary trap is contaminated	Clean and condition trap. See Chapter 4, Molecular Sieve Trap Cleaning
"W" shaped or negative inflection on, CO_2 peak (TIC, TOC or POC)	Gross CO_2 contamination in Purge and Carrier Gases	Change gas cylinder. Use gas with 99.98% + purity
Purge valve remains on or off continually	Faulty PC board or cable	Follow procedure in Chapter 5, Keypad Functions, to determine exact solution
Sample pump does not rotate	Faulty PC board or cable	Follow procedures in Chapter 5, +120 volt Pump Control Check and Keypad Functions, to determine exact solution
Acid pump does not dispense acid	Faulty PC board or cable	Perform Keypad Fuctions test in Chapter 5 to determine exact solution
Oxidant pump does not dispense oxidant	Faulty PC board or cable	Perform Keypad Fuctions test in Chapter 5 to determine exact solution
Fan does not rotate when trap is cooling	Faulty PC board or cable	Perform Keypad Fuctions test in Chapter 5 to determine exact solution

SYMPTOM	PROBABLE CAUSE	CORRECTIVE ACTION
Incorrect temperature reading is displayed	Faulty PC board or cable	Check Temperature Control Circuit as described in Chapter 5 to determine exact solution
	Open thermocouple circuit	Check thermocouple junction and MUX board terminals for loose connections
Zero mass and concentration displayed/printed with non-zero millivolt reading Water dripping from injection port	No calibration data	Enter calibration data (see Chapter 4)
IR baseline zero too high (greater high 150 mV with no adjustment)	Leaky septum	Replace septum as outlined in Chapter 5, Changing Injection Port Septum
	Contaminated IR sample cell	See IR Cell Maintenance in Chapter 5
	Digestion vessel heater too hot	Check heater 3 temperature setting and set to 95-100°C
	Purge gas flow too high	Adjust flow to proper setting as outlined in Chapter 5
	Digestion vessel drain tube not positioned properly - insufficient draining of vessel	See Digestion Vessel Maintenance in Chapter 5
	Total volume of acid + oxidant + sample exceeds volume of vessel	Total volume should be adjusted to no more than 16 ml

ERROR Messages

TIC = *ERROR* ppmC
POC = *ERROR* ppmC
POC = *ERROR* ppmC
TC = *ERROR* ppmC
POX = *ERROR* ppmX

If any of these messages appear, either on the display board or printed, then sample volume has been set to zero. Setting sample volume to a non-zero number as outlined in **Chapter 2** should correct this error.

WARN: LINEARITY ERR

This message as seen on the intruments' display screen indicates that the infared signal, as monitored in IR OUTPUT mode or a strip chart recorder, has exceeded 1000 mV, and that the calculated concentration is an extraploated value. The message is intended to warn the analyst that one of two conditions exists:

- The carbon mass detected by the analyzer has exceeded the 50 ug C mass range and that the sample should be either diluted and re-analyzed or a smaller sampler volume should be used to keep the carbon mass within the 50 ug C range.

- The IR baseline is too high. This condition is evident if a sample that is known to have less than 50 ug C still gives this error message. Follow the procedure in Chapter 5 for adjust IR Zero to correct this problem.

N.L. (Non-Linear) ERROR on Printer Output

Example: SPL #---- HH:MM:SS N.L. TIC = ---- mV

This error message indicates the same condition(s) mentioned above and corrected in the same manner. It is noted here that the N.L. error may appear on the printer output even when a 1000 mV output is not exceeded. This would be the case when a N.L. condition occurred in a previous analysis and the Model 700 was in a display mode other than NORMAL. To correct this printer condition, simply put the Model 700 in the NORMAL display mode for subsequent analyses.

WARN: IR SATURATED

This message as seen on the instrument display screen indicates that the infrared signal, as monitored by IR OUTPUT mode or a strip chart recorder, has exceeded 1600 mV, and that the calculated concentration falls outside the statistical extrapolation range of the linerization coefficients for the detector. The message is intended to warn the analyst that one of two conditions exists:

- The carbon mass for that sample exceeds the linear detection limit of the IR and that the sample should be either diluted and reanalyzed or a smaller sampler volume should be used to keep the carbon mass within the 50 ug C range.

- The IR baseline is too high. This condition is evident if a smaple that is known to have less than ca. 75 ug C still gives this error message. Follow the procedure outlined for adjusting IR Zero in Chapter 5 to correct this problem.

O.R. (Over- Range) ERROR on Printer Output

Example: SPL #---- HH:MM:SS O.R. TIC ----mV

This error message indicates the same condition(s) mentioned above and corrected in the same manner. It is noted here, however, that the O.R. error may appear on the printer output even when a 1600 mV output is not exceeded. This would be the case when an O.R. condition occurred in a previous analysis and the Model 700 was in display mode other than NORMAL. To correct this printer condition, simply put the Model 700 in the NORMAL display mode for subsequent analyses.

Needle Sample Sparge Tube
2 Part # 173667 @$17.00

Condensation Chamber Part# 166109

Chapter 7
Replacement Parts

In **Chapter 2**, the various components of the 700 were identified and named. This chapter is simply a listing of the order numbers for these components and for other replacement parts and support items. Replacement parts which we consider expendable are marked as such. Expendable components are ones which are to be replaced regularly or are easily broken or deformed.

Parts for 700

PART NAME	PART #	U/M	XPND
Miscellaneous			
Bottle - Reagent (500 ml)	169153	ea	*
Condensation Chamber	166109	ea	*
Digestion Vessel	192039	ea	*
Digestion Vessel Cap	166084	ea	
Fan - Radial Cooling	141359	ea	
Filter - In-Line 10 m (reagent bottle)	182246	ea	*
Filter Assy-Gas/Liquid Separator	192120	ea	*
Flowmeter Assy	161571	ea	
Injection Block	166232	ea	
O-Ring - SIL 3/8 x 1/16	192047	ea	*
O-Ring - SIL 3/16 x 1/16in	166167	ea	*
O-Ring - SIL 3/4 x 1/16in	166175	ea	*
O-Ring - SIL Wafertoc 4in	167959	ea	*
O-Ring - VTN Reagent Pump Poppet	178822	ea	*
O-Ring - VTN Reagent Pump Seal	178814	ea	*
Power Cord	116038	ea	
Power Supply - 12 V, 3.4A	166547	ea	
Power Supply - 5V, 6A	166539	ea	
Pump - Reagent Metering	182250	ea	
Pump - Reagent Metering - Rebuilt (req. exchg)	182816	ea	
Screw - Mach BH SS 4-40 x 1/4	132001	ea	*
Spacer - P.P., Condensation Chamber	192054	ea	*
Thermocouple - Type K 20 Ga 33 in	168238	ea	
Trap - Charcoal 1/8 OD	160375	ea	*
Trap - Molecular Sieve 1/8 OD	167149	ea	*
Tube Assy - Permeation	189829	ea	*
Tubing - TFE 1/16 x .010 ID	139065	ft	*
Tubing - TFE 1/16 x .031 ID	145591	ft	*
Tubing - TFE 1/8 x .063 ID	147901	ft	*
Tubing - Tygon 1/16 OD	111260	ft	*
Tubing - Urethane 1/8 x 1/16 Clear	166224	ft	*
Valve - Actuator Gas Solenoid	166349	ea	
Valve - Check	182238	ea	*
Valve - Purge Gas Solenoid	165797	ea	
Wire - Platinum	165581	ft	*

PART NAME	PART #	U/M	XPND
Boards			
AC Power Control Board	165028	ea	
AC Power Control Board - Rebuilt (req. exchg)	182766	ea	
Display Board	164989	ea	
Display Board - Rebuilt (req. exchg)	182774	ea	
Fuse - A/C Power Board (F1) 4A Slow Blow	119339	ea	*
Fuse - A/C Power Board (F2) 6 1/4A Slow Blow	170449	ea	*
I/O Board	165060	ea	
I/O Board - Rebuilt (req. exchg)	182782	ea	
Processor Board	165044	ea	
Processor Board - Rebuilt (req. exchg)	182808	ea	
Software - (EPROM)	173295	ea	
Thermocouple MUX Board	165002	ea	
Thermocouple MUX Board - Rebuilt (req.exchg)	182824	ea	
Fittings			
Adapt BR 1/4MNPT x 1/8 Tube	152215	ea	
Adapt F-Luer TZL 1/4-28	166266	ea	*
Adapt M-Luer Lock 1/4-28	110791	ea	*
Adapt PP 1/4-28 x 1/4 Barb	170994	ea	*
Adapt PPF - Luer 1/16 Barb	192112	ea	*
Adapt PPM - Luer 1/16 Barb	192104	ea	*
Adapt TZL M-Luer 1/4-28	166258	ea	*
Coupling PP 1/4-28	166274	ea	*
Frl 1/8 Br SW	128082	ea	
Frl 1/8 SS SW	149452	ea	
Frl TFE 1/4 - Condensation Chamber	175978	ea	
Frl TFE 6 mm - POC Furnace Tube	137853	ea	
Injection Port PE 1/4-28	166828	ea	
Nut 1/8 Br SW	128108	ea	
Nut 1/8 SS SW	169773	ea	
Nut 1/4 SS - Condensation Chamber	178574	ea	
Nut Kel-F 1/16 Male W/Frl	168337	ea	
Plug TFZ 1/4-28	166430	ea	*
Tee BR 1/8 Tube	124750	ea	
Tee PTFE 1.5 mm ID	170431	ea	*
Tube End PP 1/4-28 x l/16 Blue	166331	ea	*
Tube End PP 1/4-28 x 1/16 Black	166323	ea	*
Tube End PP 1/4-28 x 1/16 Clear	166282	ea	*
Tube End PP 1/4-28 x 1/16 Red	166307	ea	*
Tube End PP 1/4-28 x 1/16 Yellow	166315	ea	*
Tube End PP 1/4-28 x 1/8 Blue	166381	ea	*
Tube End PP 1/4-28 x 1/8 Green	166357	ea	*
Tube End PP 1/4-28 x 1/8 Black	166399	ea	*
Tube End PP 1/4-28 x 1/8 Clear	165862	ea	*
Tube End PP 1/4-28 x 1/8 Red	166365	ea	*
Tube End PP 1/4-28 x 1/8 Yellow	166373	ea	*
Union 1/8 SW - POC Furnace Tube	124768	ea	
Union SS 3/16 - 1/8 Tube	171009	ea	
Union SS 1/4 - 1/8 Condensation Chamber	124735	ea	

PART NAME	PART #	U/M	XPND
Valves			
Stator - 6 Port Rotary Valve 1/16 in	172750	ea	*
Stator - 6 Port Rotary Valve 1/8 in	172700	ea	*
Valve - 6 Port TFE 1/16 in	171976	ea	
Valve - 6 Port TFE 1/8 in	183731	ea	
Valve - Actuator Gas Solenoid	166349	ea	
Valve - Reagent Check	182238	ea	*
Valve - Purge Gas Solenoid	165797	ea	
Infrared Detector			
Cell Assy - Infrared Detector	178749	ea	
Detector - NDIR w/Encl - Rebuilt (req. exchg)	182832	ea	
Detector - NDIR w/Enclosure	171885	ea	
Foil - IR Cell, Gold-Plated	173279	ea	*
Fuse - 0.5 Amp	115469	ea	*
Window - IR Cell 20 mm	173287	ea	*

Parts for Options

Process Sampling

Ftng - Adapt PP 1/4-28 x 1/4 Barb	170994	ea	*
Ftng - Adapt TZL M-Luer 1/4-28	166258	ea	*
Option - Process Sampling	164559	ea	
Option - Process Splg Multiplexed	169088	ea	
Option - Small Loop Sampling	172784	ea	
Plate - Sample Pump Adapter	178764	ea	
Pump - Sample Peristaltic 75 ml/min	178830	ea	
Sample Loop - 1 ml	166498	ea	*
Sample Loop - 10 ml	166513	ea	*
Sample loop - 100 ml (microliter option only)	172734	ea	*
Sample loop - 40 ml (microliter option only)	172726	ea	*
Sample Loop - 5 ml	166505	ea	*
Sample loop - 500 ml (microliter option only)	172718	ea	*
Tube - SIL 3/16 ID, Sample Pump (75ml/min)	177247	ea	*

POC/POX

Chem - POC Furnace Catalyst	154740	ea	*
Fiber - Qtz Wool POC Furnace Tube 1/2 oz	144501	ea	
Ftng - FRL TFE 6 mm - POC Furnace Tube	137853	ea	
Ftng - Union 1/8 SW - POC Furnace Tube	124768	ea	
Option - Purgeable Organic Carbon	164533	ea	
Option - Purgeable Organic Halides	174227	ea	
Reactor - 4420, POX	169955	ea	
Reactor - 4420 (Rebuilt), POX	179580	ea	
Trap - POC/POX Packed 1/8 OD	168197	ea	*
Tube - Ni Reaction (6 pack) POX	170514	ea	*
Tube - Qtz Furnace Packed 6 mm	168189	ea	*
Tube - Qtz Furnace Unpacked 6 mm	111773	ea	*

PART NAME	PART #	U/M	XPND
Autosampler			
Collar - Hold Down Foot, Septum Piercing	174029	ea	
Collet Assy - ASM	179283	ea	
Foot - Hold Down, Septum Piercing	174037	ea	
Kit - Septum Piercing	178046	ea	
Module - Autosampling	168402	ea	
Module - Autosampling - Rebuilt (req. exchg)	182675	ea	
Needle - Septum Piercing 3.5 in 15 Ga	173980	ea	*
Needle - Septum Piercing 7 in 18 Ga	178723	ea	*
Option - Autosampling 14 ml 76 Spl	169012-16	ea	
Option - Autosampling 40 ml 27 Spl	169012-28	ea	
Option - Septum Piercing 40 ml	173328-28	ea	
Rack - ASM 16 mm Tube 20/Set	173170	ea	
Rack - ASM 18 mm Tube 20/Set	174235	ea	
Rack - ASM 28 mm Tube 10/Set	173162	ea	
Rod - SS Hold Down Foot, Septum Piercing	174011	ea	
Rod - SS Pipet ASM (Threaded)	178368	ea	
Screw Cap - Open Hole for 40 ml	168147	ea	*
Screw Caps - Open Hole for 14 ml (50/pk)	174558	pk	*
Screw Caps - Open Hole for 40 ml (50/pk)	174532	pk	*
Septa - Teflon Faced for 14 ml (50/pk)	169054	pk	*
Septa - Teflon Faced for 40 ml (50/pk)	173211	pk	*
Septum - 18 mm x .095 TFE/SIL	166092	ea	*
Septum - 22 mm x .095 TFE/SIL	168155	ea	*
Septum - 5 mm x .125 TFE/SIL	166133	ea	*
Spacer - ASM Wash Station	178707	ea	
Vessel - Pyrex Wash ASM	178731	ea	*
Vials - Autosampler 14 ml (344/bx)	169038	bx	*
Vials - Autosampler 40 ml (100/bx)	173196	bx	*
Data Handling			
Cable - Printer Interface	168923	ea	
Option - DHS-7 Software	178996	ea	
Option - DHS-7 System I	178988	ea	
Option - DHS-7 System II	179390	ea	
Option - DHS-7 System III	179408	ea	
Option - DHS-7 System III+	179416	ea	
Option - Printer - Dot Matrix 80 Col	169004	ea	
Option - Serial Communications RS232	169070	ea	
Option - Strip Chart Recorder	172487	ea	
Paper - Printer (400 Sheets)	138546	pk	*
Paper - Recorder (6/pk)	172501	pk	*
Pens - Recorder (6/pk)	172510	pk	*
Ribbon - Printer (Epson)	139502	ea	*
Ribbon - Printer (Okidata)	178871	ea	*
Ampule TOC			
Adaptor - Ampule collar support	124453	ea	*
Ampules - Precombusted (200/bx)	130021	bx	*
Chem - Cupric Oxide Powder (500 g)	136946	ea	*
Chem - Cupric Oxide Pre-Combusted (50g)	169319	ea	*
Cutter Plunger	124255	ea	

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PART NAME	PART #	U/M	XPND
Dipper - Persulfate/CuO measure	110114	ea	*
FTNG - Union SS 1/4 - 1/8, Cutter Plunger	124735	ea	
Kit - Ampule Breaking Assy.	169210	ea	
Manual - P&S Operating Procedures	176653	ea	
Module - Purging and Sealing	132894	ea	
Option - Ampule Solids	164541	ea	
O-Ring - Cutter Plunger	116350	ea	*
O-Ring - Latex Ampule Mounting Seal	116186	ea	*
Receiver Assy - Ampule	124339	ea	
Septum - Orange	116194	ea	*
Tube - Glass Cutter Plunger Barrel	124248	ea	
Vial - Standardization	124412	ea	*

Needle Sample Sparge 21.6"cm Part # 173667 $17.00

Wafertoc

Ampules - Precombusted (200/bx)	130021	bx	*
Chamber - Inner Back 4in Wafer	167040	ea	
Chamber - Inner Front 4in Wafer	167032	ea	
Chamber/Block - 4in Wafer	164179	ea	
Chamber/Block - 6in Wafer	164187	ea	
Heater Block - Wafertoc	164161	ea	
Option - Wafertoc 4-inch	169096	ea	
Option - Wafertoc 6-inch	174946	ea	

Other Parts

Accessories

Chem - Ascarite (500 g) - Refill for Ascarite Scrubber	110122	ea	*
Chem - Phosphoric Acid (500 ml)	110080	ea	*
Chem - Potassium Biphthalate (500 g)	136954	ea	*
Chem - Sodium Carbonate, Anhydrous (500 g)	136962	ea	*
Chem - Sodium Persulfate (100 g)	174194	ea	*
Chem - Sodium Persulfate (500 g)	173518	ea	*
Installation by factory service rep	141531	ea	
Manual - Operating Procedures Model 700	168519	ea	
Option - Voltage Convertor 100 VAC	169187	ea	
Option - Voltage Convertor 230 VAC	169179	ea	
Reagent - Phosphoric Acid, Cleaned (1 ltr)	169244	ea	*
Reagent - Sodium Persulfate, Cleaned (1 ltr)	169236	ea	*
Reagent - Standard Carbonate 1000 ppmC (10ml)	169294	ea	*
Reagent - Standard KHP 1000 ppmC (10 ml)	169252	ea	*
Reagent - Water, Ultrapure in TIC/TOC (1 ltr)	169301	ea	*
Regulator - Helium/Nitrogen (Specify gas)	155417	ea	
Regulator - Oxygen	150326	ea	
Snoop - Squirt Bottle	129767	ea	*
Syringe - 10 ml x 2in Needle	169335	ea	*
Syringe - 10 ml x 2in Needle	167545	ea	*
Syringe - 100 ml x 2in Needle	110221	ea	*
Syringe - 2.5 ml x 2in Needle	137051	ea	*
Syringe - 25 ml x 2in Needle	110205	ea	*
Syringe - 5 ml x 2in Needle	169327	ea	*
Syringe - 50 ml x 2in Needle	110171	ea	*
Syringe - 500 ml x 2in Needle	137069	ea	*

PART NAME	PART #	U/M	XPND
Tool - Allen Wrench-2.5 mm	171926	ea	*
Tool - Flaring Tip 1/16 in Tube	169137	ea	*
Tool - Flaring Tip 1/8 in Tube	169129	ea	*
Tool - Soldering Iron 40 W	168808	ea	*
Tool - Wrench 1/4 x 5/16 Open-End	169161	ea	*
Tube - Scrubber Ascarite 120 ml	169145	ea	*

Kits

Kit - Check Valve/Filter - Reagent Pump	182253	ea
Kit - Hook-Up	171215	ea
Kit - Maintenance Accessories	168816	ea
Kit - Permeation Tube	196469	ea
Kit - Reagent Pump Rebuilding	178806	ea
Kit - Septum Piercing	178046	ea
Kit - Service 700 Domestic	169111	ea

Options

Option - Ampule Solids	164541	ea
Option - Autosampling 14 m 76 Spl	169012-16	ea
Option - Autosampling 40 m 27 Spl	169012-28	ea
Option - DHS-7 Software	178996	ea
Option - DHS-7 System I	178988	ea
Option - DHS-7 System II	179390	ea
Option - DHS-7 System III	179408	ea
Option - DHS-7 System III+	179416	ea
Option - Printer - Dot Matrix 80 Col	169004	ea
Option - Process Sampling	164559	ea
Option - Process Splg Multiplexed	169088	ea
Option - Purgeable Organic Carbon	164533	ea
Option - Purgeable Organic Halides	174227	ea
Option - Remote Bay	174350	ea
Option - Septum Piercing 14 ml	173328-16	ea
Option - Septum Piercing 40 ml	173328-28	ea
Option - Serial Communications RS232	169070	ea
Option - Small Loop Sampling	172784	ea
Option - Strip Chart Recorder	172487	ea
Option - Voltage Convertor 100 VAC	169187	ea
Option - Voltage Convertor 230 VAC	169179	ea
Option - Wafertoc 4-inch	169096	ea
Option - Wafertoc 6-inch	174946	ea

Chapter 8
Major Optional Equipment

This chapter outlines the conceptual and operating aspects of some major optional equipment which is available for use with the Model 700, but is not needed by the majority of users. The equipment discussed in this chapter includes the:

- Purgeable Organic Halide Option (POX)
- DHS-7 Data Handling System
- Process Sample Multiplexer

Purgeable Organic Halides Option (POX)

Overview: The Purgeable Organic Halide (POX) option for the Model 700 TOC Analyzer is a feature which allows for quantitative monitoring of volatile organic halides in water samples. The system is comprised of the Purgeable Organic Carbon (POC) option of the Model 700 and O.I. Analytical's Model 4420 Electrolytic Conductivity Detector (ELCD). Results are displayed in parts-per-million halide.

The methodology for the POX option is consistent with the EPA protocol for the analysis of volatile halides using purge and trap without GC separation. This method and the one described here in this procedure uses chloroform as the reference compound and concentrations are those based on detector response for chloride.

Safety: The toxicity or potential health hazard of chemicals used in this method has not been precisely defined. However, all chemicals used, should be treated as a potential health risk and exposure to the chemicals should be minimized. Each laboratory is responsible for maintaining awareness of OSHA regulations regarding safe handling of chemicals and associated equipment used in this method.

Chloroform has been identified as a suspected cancer causing agent in laboratory animals and humans. Pure material and stock standard solutions of this compound should be handled under an OSHA approved fume hood. Appropriate skin protection and approved toxic gas respirator should be worn when the analyst handles high concentrations of this toxic substance.

Reagents and Materials: The items required for the POX option differ slightly than those used for TIC/POC/TOC analysis on the Model 700. The following list is the minimum requirement of parts not supplied with the instrument for installation and operation of the POX option.

Reagent Water: Deionized or degassed water - water may be degassed by boiling in a suitable container for 1 hour.

Chloroform - 99.99+% purity

n-Propyl Alcohol - ACS reagent grade or better

Gas Service

- Type - Helium and Hydrogen
- Purity 99.99+% (zero grade)

Regulators - Two stage, Stainless Steel diaphram suitable for 30 psig (207 kPa) delivery pressure. One for helium gas supply and one for hydrogen gas supply.

Gas Supply Tubing - 1/8" OD x 0.065" ID Stainless Steel, copper or teflon of suitable length to reach instrument from gas supply regulator.

Initial Set-Up: Before attempting the set-up and operation of the POX option, the analyst should be familiar with the operation of both the Model 700 TOC Analyzer and the Model 4420 ELCD as outlined in each of the operator manuals for the two instruments.

- After installation of the Model 700 has been completed as outlined in **Chapter 3** of this manual, connect a supply of pure helium to the two gas inlet bulkheads. The actuator gas may be from a separate cylinder of air, nitrogen or other nonflammable gas or may be provided to the inlet using a tee from the helium gas line.

- Carefully unscrew the 4420 reactor from the detector base and remove the 1/8" to 1/16" graphite reducing ferrule from the reactor base.

It will be necessary to remove the left bay covcer to complete installation (see Chapter 3).

WARNING: If POC analysis have be run previous to POX setup, turn off oxygen supply at the source as hydrogen will be used for POX analysis and could result in a potentially hazardous gas mixture.

- Mount the 4420 reactor base on the rear of the left bay of the Model 700 as shown in Fig. 9.1 using two 8/32" x 3/4" screws, lock washers, nuts, and a piece of ceramic wool (2"x2") as an insulator between the reactor base and back wall.

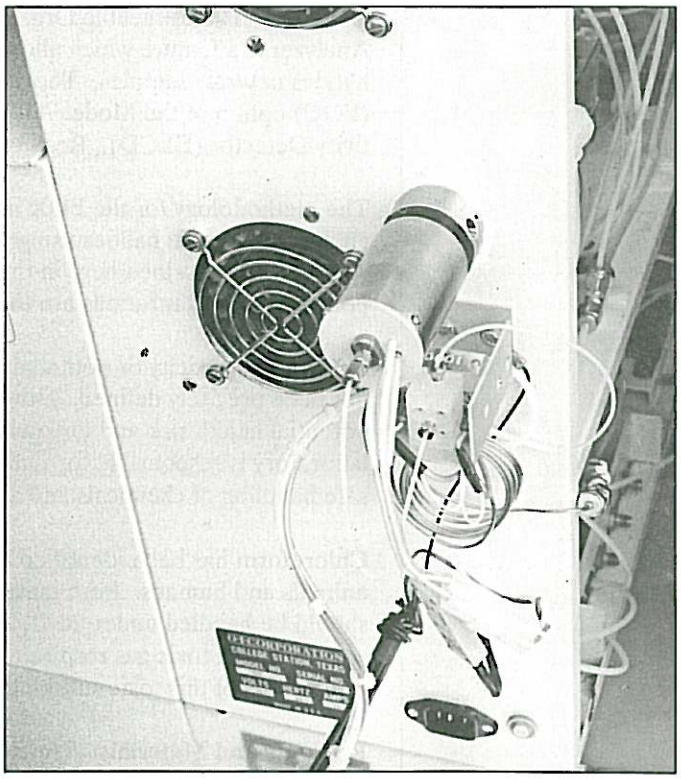


Fig. 8.1 700 POX Option Sub-Assembly

- Mount the detector cell horizontally with the 1/8" solvent return line toward the back of the 700 chassis using two 4/40" x 3/8" screws, lockwashers and nuts.

- Carefully screw the reactor (with reaction tube and reducing ferrule) into the detector base. Tighten the reactor to a gas tight seal with finger pressure only. Hold the reactor by the metal body and do not twist the ceramic cap during this operation.

- Route the gas lines and electrical connections to the rear of the 4420 and complete hook-up in accordance to Fig.s 3.2 and 3.3 of the 4420 operators manual.

- Connect the hydrogen restrictor to the reaction gas inlet tube. The reaction gas inlet tube is the long 1/16" OD tube on the reactor base.

- Connect the other end of the restrictor to the hydrogen gas source. The gas should be of high purity to obtain optimum results.

- Connect the 1/16" teflon tube from the conductivity cell to the 1/16" union on top of the reactor using the 1/16" graphite ferrules supplied.

- Locate the POX external interface cable (OI Part #180-018).

Do not turn on hydrogen at this time.

- Connect one end of the white lead to the 1 volt output on the rear of the 4420 module and the other end to T1 on the 700 terminal strip (see Fig. 8.2).

WARNING: Do not overtighten the fitting.

- Connect one end of the black lead to the common output on the rear of the 4420 Module and the other end to T2 on the 700 terminal strip.

- Connect one end of the clear lead to the ground terminal on the rear of the 4420 Module and the other end to either of the two terminal strip retaining screws located adjacent to T2.

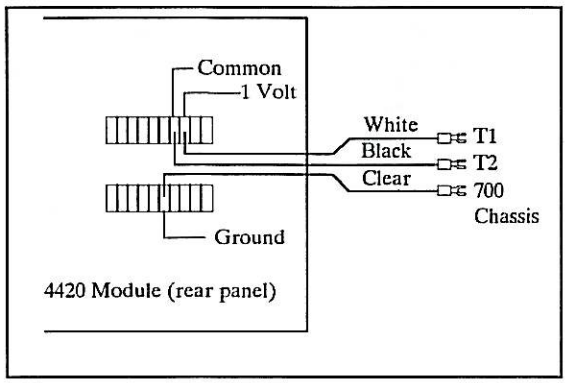


Fig. 8.2 POX External Interface Cable Connections

- Disconnect the tube end fitting from port 5 of the POC valve (refer to the POX plumbing schematic in Chapter 9) and plug the tee with the TFZ tube end plug (OI Part #166-430) provided in the POX Startup Kit.

- Connect a source of dry inert gas (N_2 or He) to the auxiliary bulkhead fitting (same as for oxygen when in POC mode). This gas will provide the counter current gas for the permeation drying tube and is necessary for proper operation of the tube.

The same helium gas supply used for the sample purge gas may be used for this counter current gas flow. Thus, a single helium gas supply could possibly be used to provide purge, actuator and counter current gas flows by using a cross connector.

- Connect the 1/16" x 0.010" ID teflon gas line (provided in the POX Startup Kit) to the back of the reactor base and port 5 of the POC valve.

WARNING: n-Propyl alcohol is a flammable liquid. Keep sparks and open flames away from the solvent reservoir and use in an adequately ventilated area.

- Add enough n-propyl alcohol to the solvent reservoir to fill it at least half full. Close the lid tightly when complete.

- Replace the left bay cover of the Model 700 and place the 4420 Module to the left side of the Model 700.

Setting Flow Rates

- Turn on the helium, Actuator gas, and auxillary gas (if on separate lines) and set regulator(s) for 30 psi (207 kPa) delivery pressure. Confirm purge in reagent bottles of the Model 700. If a separate auxilliary gas is being used for the per meation tube, counter current turn on this gas supply and set the pressure to 30 psi (207 kPa).

- Turn on the hydrogen reaction gas and set regulator to 30 psi (207 kPa) delivery. Check all connectors for gas leaks using a leak detection solution.
- Power up the Model 700 according to **Chapter 4** of this manual.
- Enable the POX Option by pressing the **SET SYSTEM CONFIGURATION** key (POX Option is a Group 3 option).
- Select Manual mode by pressing the **SELECT ANALYSIS MODE** key.

CAUTION: Hydrogen is flammable; keep open flames and sparks away from detector.

- Turn on Purge Valve by pressing **#9** on the key pad panel. Confirm DISPLAY shows PV> and set Purge flow to 6 on rotometer (approximately 110 cc/min as measured at Sample Drain Line).
- Place the POC Valve in-line with the 4420 by pressing **#2** on the key pad panel. Confirm DISPLAY shows TV2> and set Detector flow to 2 on rotometer (approximately 35 cc/min as measured through 0.010" ID teflon tube from port 5 of POC trap valve).
- Select POC/TIC/TOC analysis by pressing the **SELECT ANALYSIS MODE** key.
- Power up the Model 4420 in accordance to Chapter 4 of the operators manual.
- Turn on reactor and set reactor temperature to 850° - 925°C.
- Turn on the solvent pump and set solvent flow to approximately 50 ul - 100 ul/min as measured from the solvent return line.

This is approximately 4 turns clockwise from the full counterclockwise position of the solvent control knob.

- Confirm that detector selection switch on rear of Model 700 is set to measure the 4420 ELCD response (DOWN).
- Follow the instructions outlined in **Chapter 4** for Sample Introduction and Running Reagent Blanks in this operators manual for analysis of samples.

WARNING: Do not disconnect gas lines from Model 4420 when solvent pump is on as the electrolyte may back up into the nickel reaction tube causing deactivation of catalytic surface and a possible fire.

Calibration

Concept: The measurement of POX is similar to that for POC (see **Chapter 4** of Model 700 operators manual) except that the Model 4420 ELCD response is due to a mass of hydrogen halide (see Chapter 1 of the Model 4420 operators manual).

Chloroform has been established as a reference compound for the efficiency of the purge and trap analysis. Thus, it is used here as a basis for determining percent recovery and detector calibration. However, since as of now there is no established procedure for POX standards, any purgeable organic halide may be used as a laboratory reference compound.

Purge times for the analysis should be set to obtain maximum recovery of the compound(s) of interest for calibration. For chloroform, the purge flow as set using the procedure above and the purge time in a normal POC-Only analysis is sufficient for sample volumes of 1.0 cc or less. For a sample volume of 5 cc or greater it is suggested that a 3.5 minute extended purge be set in the SET TIMES function.

Standards should be prepared as parts-per-million halide (e.g. chloride). For a 100 ppm Cl chloroform, dissolve 7.6 ul of neat chloroform into 100 ml of absolute methyl alcohol. Standards can then be made by using microliter dilutions into appropriate volumes of deionized or degassed reagent water.

Do not use this methyl alcohol solution as a primary standard but only as a stock solution for water dilution standards. Running the methyl alcohol directly will cause irreversible damage to the nickel reaction tube.

Procedure: Calibration for POX follows the same general scheme as those described in Chapter 4 of this manual (i.e. blank determination and single point calibration) with the following noted conditions.

P/T Mode: Linearity of the Model 4420 in the P/T mode under the conditions of analysis described earlier in this chapter allow measurement of chlorine from 5 ug Cl to 30 ug Cl. Thus sample volume should be chosen to allow for this range of linearity. If for example, a 10 ml sample is being analyzed the lower detection limit would be 500 ppb Cl. If a 0.3 ml sample is being used, the upper detection limit is 30 ug Cl or 100 ppm Cl.

Nitrogen (N) Mode: Linearity of the Model 4420 in the N mode under the conditions of analysis described earlier in this chapter, allow measurement of chloride from 10 ng Cl to 3.0 ug Cl. Thus, if a 10 ml sample is being analyzed the lower detection limit is 1.0 ppb Cl, or if a 0.3 ml sample is being analyzed, the upper detection limit is 10 ppm Cl.

TOC Reconfiguration

- Power down the Model 700.
- Turn off the reactor and solvent pump on the Model 4420 and then power down.
- Turn off hydrogen gas supply.
- Remove left bay cover of Model 700 as described in **Chapter 3.**
- Disconnect the 1/16" x 0.010" teflon gas line from Port 5 of the POC valve.
- Remove the TFZ tube end plug from the oxygen inlet tee and place it on the 1/16" x 0.010" teflon tubing connected to the 4420 reactor base.
- Connect the tube end fitting from Port 5 of the POC valve to the oxygen inlet tee leading to the furnace, and reconnect the auxiliary bulkhead fitting to an oxygen supply. Turn on oxygen supply.
- Replace left bay cover.
- Set detector selection switch on rear of the Model 700 to measure infrared detector response (UP).
- Proceed with carbon analysis per instructions in **Chapter 4** of this manual.

If purge gas or actuator gas other than helium is to be used for TOC analysis, it should also be reconnected at this time per Chapter 3.

DHS-7 Data Handling System

The user is advised to read the entire contents of this section before performing the startup of the DHS-7 software or collecting of data to prevent loss of data.

Overview: The DHS-7, Data Handling Software - 700 consists of a Model 700 Total Organic Carbon analyzer, a statistical analysis software package and a PC compatible computer.

The Model 700 TOC together with the Data Handling Software (DHS) and a computer present the labratory analyst with a complete sample to report system. The results of the Model 700 sample analysis is captured by the software package via the computer. The data collected can than be labeled, have statistical analysis preformed and reports generated.

The DHS comes on two diskettes. One is a set of programs for limited memory (256K) labeled DHS-7 LMT and the other is for expanded memory (512K and up) labeled DHS-7 EXP. The limited version is black and white while the expanded version is in color. Both versions appear the same on a monochrome monitor. In either case the user is requested to follow these instructions before using the DHS package.

Users of two floppy disk computers: Start your computer with your DOS diskette in drive A. Insert a blank diskette in drive B and type FORMAT B:/S. This instructs your computer to format the diskette in drive B and to include the hidden system files. Upon completion of the format process type COPY COMMAND.COM B: This will copy the DOS program to drive B enabling the use of the auto start-up feature. After the computer responds with 1 file copied, insert the appropriate DHS diskette (DHS-7 EXP or DHS-7 LMT) in drive A and type FLOPCOPY. This will copy all files to drive B. After the copy is complete place the working copy from drive B in drive A and store the original. This working copy can now be used to start your computer and it will contain the DHS. A formatted file diskette will be needed for drive B.

Users of hard disk computers:

After computer initialization, insert the appropriate DHS diskette (DHS-7 EXP or DHS-7 LMT) in drive A and type HARDCOPY [path]. If a path is used the format must follow this example: HARDCOPY MYSUBDIR/[/MORESUBS]. If a path is not supplied all files will be copied to the main(root) directory of drive C. The user has the opportunity to copy the CONFIG.SYS file or enter the following two lines in an existing CONFIG.SYS file:

```
BUFFERS = 5
  FILES = 24
```

The computer must then be restarted. Type [/MYSUB/][/MORSUBS/] GO to start the DHS. For auto-startup place the command GO [/MYSUB][/MORSUBS] as the last line in the AUTOEXEC.BAT file. If an AUTOEXEC.BAT file does not exist in the main(root) directory of drive C one may be created by typing the following lines:

```
COPY CON: AUTOEXEC.BAT <enter>
GO [/MYSUB/][/MORSUBS/] <enter>
Then press the <F6> key and <enter>
```

The automatic start up program is now complete. Restart the computer and follow the prompts.

Background Data Collection program: The user may start the data collection program, DATATRAP for expanded memory (512K and up) or DATATRPL for limited memory (256K), by using the following syntax:

WARNING: Plugging the serial cable into any connctor other than the RS-232 connector (i.e. External Control, Printer, etc), may cause damage to the computer or the Model 700 electronics.

DATATRAP [d:][path] [filename][ext] [com port]
or DATATRPL [d:][path] [filename][ext] [com port]

See the next section, DATATRAP for more details.

DHS software: The Data Handling Software may be initialized by typing DHS. The Main Screen will be presented.

General Operating Procedures

- Plug one end of the serial cable supplied with the DHS-7 software package into the RS-232 connector on the Model 700 (see **Chapter 2** for location on rear of instrument). Plug the other end into com port 1 (preferably) or Com Port 2 on the computer. Users of 'AT' style computers may need to acquire an adaptor cable to use the com ports.

- Configure the Model 700 TOC before starting the DATATRAP data collection program. Changing any method parameters will disable the data collection program. To be assured of data collection, the user will see the disk drive pilot lamp light when the 700 TOC displays the computed analysis on the display panel.

- Never press the print button while the data collection program is collecting data. If this occurs, the user may delete the lines of information by using a word processing program and removing only those lines.

- The printer cable supplied is used to connect the computer to the printer (usually supplied with the 700).

Datatrap

Technical Overview: Datatrap is a very flexible memory resident program used to collect data from a com port in the background while another program is active in the foreground (on the screen). Datatrap creates a new text file or overwrites an existing file when it is initialized.

Start-up/File creation: The complete command syntax is:

DATATRAP [d:][path][filename][.ext][com port]

The first parameter is the drive desiginator. If a drive is not specified the text file created will reside on drive A. A file path may be specified. When the path is omitted, the text file will reside in the root directory. A file name should be specified without an extension. Filenames cannot start with a number or have more than eight characters. If a filename is not specified, a file with the name OICORP.DAT will be created. If the default filename is used or a filename with an extension is used, it must be renamed to one without an extension before it can be used with the data handling software. If the filename specified exists in the directory, the original file will be overwritten.

A com port may be designated by including a 1 or a 2 with the filespec. If a com port is not specified the program defaults to com 1. Specifying a different com port is useful when the foreground program uses a particular port. For proper operation, no two programs can share the same port.

A typical example of initializing the datatrap program would be:

DATATRAP 700DATA

In this case the text file '700DATA' would default to the root directory of drive A and the program would monitor com port 1 for incoming data. If there were no errors in setting up the paramaters, DATATRAP will respond with a 'DATATRAP loaded and memory resident' message. In the case of an error, DATATRAP will respond with a 'DATATRAP not loaded' message.

After initializing DATATRAP, the disk containing the text file may be removed another disk may be inserted. As data is received from the com port, DATATRAP will attempt to add it to the text file. If the text file is not present, the information will be stored in memory until the next time data is received. Each time data is received a disk write will be attempted. The memory can hold either one half hour (for 256k version) or 30 hours (for expanded version) of data, after which incoming data is lost. The automatic disk writing may be stopped at any time by pressing **<Ctrl>** and right shift keys. Two tones will be heard signifying program compliance. By pressing **<Ctrl>** and left shift keys the automatic disk writes are again activated and a single tone is heard. The next data received will flush the entire memory to the text file. Similarly, data capture may be stopped by using the **<Alt>** and right shift keys and restarted using the **<Alt>** and left shift keys. This function can be used when servicing the Model 700.

Another feature is closing one file and starting another after DATATRAP has been activated. Should this be desirable the user may, at the DOS prompt, enter 'DATA-TRAP [filespec]. 'DATATRAP will respond with a 'DATATRAP changed to new paramaters' message. This action can also be taken from within the DHS.

Data Handling System

Technical Overview: The Data Handling System, referred to as the DHS, is a group of files and databases that are used together with text files created by either collecting data from a Model 700 or by manually entering data from within the DHS.

Files present: The following files must be present in a single directory for the DHS to properly function:

DHS.EXE	SAMPDX.NTX	DRAWSMP.FRM
START.DBF	GRUPDX.NTX	RAWSAMP.FRM
SAMPLE.DBF	CONAME.MEM	RPRTSAMP.FRM
STATS.DBF	RAWSAMP.FRM	REPINFO.DBF
STATS.FRM		

The following files could also be present:

DATATRAP.COM	CONFIG.SYS	COMMAND.COM
GO.BAT	AUTOEXEC.BAT	

Disk drive usage: The arrangement of files on your disk drives may take any form. The general procedure for two disk systems is the DHS on drive A and text, data, and statistics files on drive B. The auto-startup and batch files noted above make these decisions for you. When a hard disk is used copy all programs and files into a single directory, preferably the main (root) directory. If the main directory is not used for the DHS, the CONFIG.SYS file must be in the main directory.

Start-up: Program initialization may take different forms. The simplest way for a two floppy drive computer is to insert the DHS in drive A and turn the computer on. The screen will show the possible options. The user may choose one of these options or initialize the program by typing DHS. When a hard disk is used, the ANSI.SYS and CONFIG.SYS files must be in the main directory.

Program Usage

Capabilities/Purpose: The DHS is designed to take the output data from the TOC analyzer or manually entered data, calculate statistical information, and generate reports and graphs. The DHS also incorporates may DOS file manipulations. Statistics and Data report formats are designed by the user.

Files created: Data files created by the DATATRAP or user input cannot include any extensions such as ????????.EXT. Data files are renamed by the DHS to include the extension of .TXT. This tells the user this file has been used at least one time by the DHS. The DHS then creates a database file with the same name but with the extension of .DBF. When statistics are calculated another file is created with the same name but with the extension of .STA. If data is collected and used there will be three files with the same name but different extensions.

Example: DATA. renamed to DATA.TXT
DATA.DBF
DATA.STA

Reports generated: The DHS can generate different kinds of reports. Two reports are considered preliminary. The first can be generated immediately after a text file is called by the DHS. This is known as the raw data report. The second is generated after the statistics have been calculated. Typically a text file (.TXT) would be used by the DHS in creating a data file (.DBF) and a statistics file (.STA). The user would the go to the Reports Screen and generate Data, Statistics and Bargraph reports.

Utilities: The Utilities Screen presents the user with file manipulation and DHS program control commands. Many standard DOS file commands are included to ease file manipulation. This screen includes the ability to create files used by other commercially available programs, to enter a company heading and to use other DHS functions.

Errors: Power loss and computer failures can occasionally occur during program execution. Great care has been taken to minimize any possible file damage. Both DATATRAP and the DHS keep files closed execpt during actual usage. Should the data file become damaged the user will need to start again with the text file. The procedure would be to delete the data file (.DBF) in question, rename the text file (.TXT) to one without an extension and regenerate the data file with the DHS. Should the statistics file become damaged the user can regenerate a new one from within the DHS. Typically, damage does not occur even when the DHS is using a file.

Special keys: The DHS program requires a minimum of user input. All program questions may be answered in upper or lower case. A few special keys are used by DATATRAP and the DHS. Key assignments for DATATRAP are given in the previous section. The **<Home>** key is generally available for use at the beginning and the end of a program function. When used after a program call, it cancels the call and returns the user to the previous menu. When the **<Home>** key is used after the call is complete, it again returns the user to the previous menu. In some places **<PgUp>**, **<PgDn>**, **< >**, and **< >** keys are used. Their function is explained on those menus. The delete key will delete characters to the right of the cursor position. The back arrow deletes characters to the left of the cursor. The **<Home>**, **<End>**, **< >**, **< >**

keys may be used for user input on the Utilities and Printer Configure menus where spaces are shown. These spaces may only show part of the required input. By using these keys the information may be scrolled horizontally. Up to thirty characters may be entered in these spaces. See Figure 1 for complete listing.

Control Keys

<Home>	Moves the cursor to the beginning of the current blank.
<Ctrl> <Home>	Moves the cursor to the beginning of the first blank.
<End>	Moves the cursor to the end of the current blank.
<Ctrl> <End>	Moves the cursor to the beginning of the last blank.
Up arrow.....	Moves the cursor to the previous blank.
Down arrow	Moves the cursor to the next blank.
Right arrow.....	Moves the cursor one character to the right.
<Ctrl> Right arrow	Moves the cursor to the beginning of the next word.
Left arrow	Moves the cursor one character to the left.
<Ctrl> Left arrow	Moves the cursor to the beginning of the current word.
<Ctrl> T	Deletes the word to the right of the cursor.
<Ctrl> Y	Deletes the rest of the field to the right of the cursor.
<Alt> C	Terminates entry if in a blank or is emergency escape if not in a blank. Always try to use 'Q' in response to the prompt. If 'Q' does not work use 'A'.
<PgUp> <PgDn>	Used to exit a blank with out an entry.
Esc	Terminates entry without saving it's value.
<Ctrl> U	Restores the current blank to it's original value.

Program Screens

View/Label/Compute Overview: The V/L/C screen is the most extensively used portion of the DHS. It is the portion of the program that calls a text file and creates a database. After a database is created the user may enter grouping information, delete unwanted samples and generate a statistics file. During this part of the process the text file is assumed to be in a main or current (for hard disk users) directory.

Call for data file: After choosing '1' from the main menu the user is presented with the following menu:

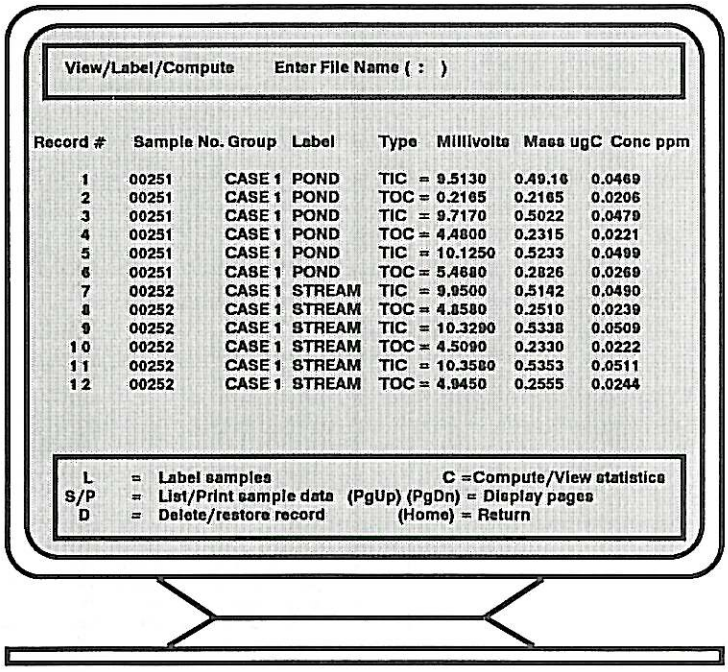
D = Data file
View/Label/Compute Enter: S = Statistics File
(Home) = Return

D = Data file: When 'D' is chosen the user is prompted for a filename. The filename may be a text file from DATATRAP or a previously created data file. A filename extension is not allowed and the file must be in the main directory of any drive or in the current directory. If a text file was called, the DHS will read the text file and create a new database. In either case the first twelve records will be displayed and a new menu will be located at the bottom of the screen.

S = Statistics file: When 'S' is chosen the user is prompted for a filename. If the drive letter is omitted the path will be used. There is no need to enter an extension. The DHS will look in the current directory or the main directory of any drive for a file with the name entered with the extension '.STA.'. The first twelve records will be displayed along with a new menu (see compute/view statistics, page XX). The data screen can be by-passed to view or create a new statistics file.

<Home> = Return: The main screen will be displayed when <Home> is used.

Data Screen Overview: The data screen presents the user with the commands needed to edit a data file including group and label name assignments. How the data file is labeled can have a significant impact on the results of computing statistics. This screen is presented:



L = Label Samples: The DHS labeler is the function used to assign 'group' and 'label' names to individual records. Once the function is initiated the following sub-menu appears:

number = start record
Label Record Number 1 (+) = Repeat last entry
(Home) = Return

The user has a set time period in which to respond. (This time period is determined via the 'Set Label speed' function found on the Utilities Screen.) Records are updated as labels are assigned.

<Enter> = display fields:

The group and label fields for record 1 will be displayed.

Record# 1 Group Name ( ) Label Name ( )

After the names are entered, the record number will be incremented and the submenu will again be displyed.

number = start record number: To select a specific starting record number to label, type the number and **<Enter>**, and that record's fields will be displayed.

<+> = repeat last entry: This function is used to copy the group and label names of the previous record and is used only for record numbers greater than 1. When this function is used the sub-menu will signify the action complete by incrementing the record number.

<Home> = Return: The data screen menu is displayed when **<Home>** is used.
S/P = List/Print Sample Data: When 'S' is chosen, pages of 12 records are displayed until the end of the file is reached. This is useful when a quick review of all the records is required. When 'P' is chosen the following sub-menu is displayed:

Note: When no response is given within the set time period, the DHS defaults to the enter response function.

D = Deleted record report
A = Active record report
<Home> = Return

D = Deleted record report: This report includes deleted records (see Delete/Restore record), displaying the word 'Deleted' at the end of the record line.

A = Active record report: This report is the same as the one described above but without deleted records present. See Fig. 8.3.

Page No. 1 Raw Data for file DATA
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REC#	SAMPLE#	GROUP	LABEL	TYPE		MILLIVOLTS	MASS ugC	CONC. ppm	TIME	DATE	DELETED
1	SPL# 00524 1			TOC	=	997.4590	50.1789	191.5220	14:16:19	09/05/86	
2	SPL# 00524 2			TOC	=	1021.1200	51.3693	196.0660	14:19:24	09/05/86	
3	SPL# 00524 3			TOC	=	1029.3100	51.7810	197.6370	14:22:29	09/05/86	
4	SPL# 00525 4			TOC	=	95.1184	4.7851	18.2637	14:25:34	09/05/86	
5	SPL# 00525 5			TOC	=	95.6970	4.8142	18.3748	14:28:39	09/05/86	
6	SPL# 00525 6			TOC	=	94.7443	4.7663	18.1919	14:31:44	09/05/86	
7	SPL# 00526 7			TOC	=	241.4070	12.1444	9.8370	14:34:49	09/05/86	
8	SPL# 00526 8		26	TOC	=	241.1090	12.1294	9.8249	14:37:54	09/05/86	
9	SPL# 00526 9		26	TOC	=	240.6420	12.1059	9.8058	14:40:59	09/05/86	
10	SPL# 00527 10		26	TOC	=	614.0610	30.8914	25.0222	14:44:04	09/05/86	
11	SPL# 00527 11			TOC	=	511.3090	25.7223	20.8352	14:47:09	09/05/86	
12	SPL# 00527 D			TOC	=	1.5721	0.0791	0.0641	14:50:14	09/05/86	
13	SPL# 00528 E		GDFH	TOC	=	1.6866	0.0848	0.0687	14:53:19	09/06/86	
14	SPL# 00528 E		GDFH	TOC	=	1.4003	0.0704	0.0571	14:56:24	09/06/86	
15	SPL# 00528 E		GDFH	TOC	=	512.4600	25.7802	20.8821	14:59:29	09/06/86	
16	SPL# 00529 F		29	TOC	=	515.1710	25.9165	20.9925	15:02:34	09/06/86	
17	SPL# 00529 F		29	TOC	=	513.9590	25.8556	20.9431	15:05:39	09/06/86	
18	SPL# 00529 F		29	TOC	=	515.4020	25.9281	21.0019	15:08:44	09/06/86	
19	SPL# 00530 G		0	TOC	=	65.0618	3.2730	0.3122	15:15:27	09/06/86	
20	SPL# 00530 G		0	TOC	=	63.0001	3.1693	0.3023	15:21:52	09/06/86	
21	SPL# 00530 H		30	TOC	=	62.5823	3.1483	0.3003	15:28:17	09/06/86	
22	SPL# 00531 H		30	TOC	=	62.2290	3.1305	0.2986	15:34:42	09/07/86	
23	SPL# 00531 I		31	TOC	=	61.3627	3.0869	0.0869	15:41:07	09/07/86	
24	SPL# 00531 I		31	TOC	=	59.7621	3.0064	0.2868	15:47:32	09/07/86	

Fig 8.3 Active Record Report

<Home> = Return: The View/Label/Compute menu will be displayed when <Home> is used.

D = Delete/Restore Record: When 'D' is chosen, the following menu is displayed:

Delete Record Number ( ) (PgUp) = Return

Record number: When a record number is entered, an asterisk denoting the that record deleted is displayed on the screen between the Record number and SAMPLE NO fields. This record will no longer be considered for statistical computations.

When the record number is again entered, the asterisk will be removed and the record will be restored to its original status.

<Home> = Return: The View/Label/Compute menu will be displayed when <Home> is used.

C = Compute/View statistics: When 'C' is chosen, the following menu is displayed:

Compute/View. C = Compute/recompute C:DATA.STA
V = View existing C:DATA.STA
(Home) = Return

C = Compute/recompute ????????.STA: When this option is chosen, the program moves to the Statistics Screen.

V = View existing ????????.STA: When this option is chosen, an existing statistics file is displayed above the Statistics Screen menu.

<Home> = Return: The View/Label/Compute menu will be displayed when <Home> is used.

<PgUp><PgDn> = Display pages: Pages of twelve records are displayed.

<Home> = Return: The Main Screen will be displayed when <Home> is used.

Statistic Screen Overview: This section of the program is used to compute sample statistics. In all cases a minimum of three like records are required to produce a sample statistics record. The 'SAMPLE NO', 'GROUP' and 'LABEL' fields from the last like record will be carried over to the statistics record. When statistics are being computed the screen also shows the mean (MEAN), standard deviation (STD DEV), and percent coefficient of variance (COE OF VAR) fields. The user has three major options and up to three sub-options. When the Statistics Screen is accessed the following menu appears:

Compute by: D = All records by date
S = Records by sample number (minimum 3 reps.)
G = Records by group name.
<Home> = Return

When computing by date **(D)**, all the samples collected each day are computed together, regardless of sample number or group name, which is useful for process operations.

When the user chooses to compute by sample number **(S)**, the DHS utilizes the sample number only. In this case a group name is not required unless a particular collection of samples is required. The group name may be located anywhere in the data file. Records with the same group name will be processed as though they were a continuous listing.

If computation by Group **(G)** is chosen, sample statistics are computed by group name only.

D = all records by Date: When 'D' is chosen the following menu is displayed:

Compute statistics by Date. A = All dates
S = Start/stop dates

A = All dates: This option allows all samples collected in a single day to be com-

puted together. More than one days worth of data may be present. Each day will be computed and displayed separately.

S = Start/stop dates: This option allows the user to select a portion of the data to be processed. The user will be prompted for a start and stop date which will be used to limit the statistical computations. Each day will be computed and displayed separately.

Compute statistics by Date.
A= All dates. Enter start date (09/05/86) (09/05/86)
S = Start/stop dates. Enter ending date (09/07/86) (/ /)

The menu shows default dates which will be used if no date or an incorrect date is entered.

S = records by sample number: When this option is chosen the following menu is presented:

Compute statistics by sample number: A = All samples
S= Start/stop samples
G= Select groups

A = All samples: This option enables all samples in the data file to be used in computing sample statistics. Each sample number must have three repetitions. Group name is not considered in computation but is included in the statistics file.

S = Start/stop samples: This option prompts the user for sample numbers to start and end with. Computations take place only on those and included sample numbers.

Compute statistics by Sample number.
A = All samples
Enter start Sample number. (326) (326)
S = Start/stop samples
Enter ending Sample number. (332) (0)
G= Select groups

This option is useful when there is a desire to limit the range of sample numbers. The 'GROUP' and 'LABEL' fields are carried over to the statistics file. Default values are given and used when either an incorrect value or **<ENTER>** is used.

G = Select groups: When this function is chosen the user is asked for group names.

Compute statistics by sample number.
A = All samples
S= Start/stop samples
Enter 1st group name. ( ) G = Select groups.

As names are entered they are checked for existence in the data file. If a name does not exist the user is prompted to enter again. A limit of 99 group names may be used. Group name specifications are ended by responding with **<ENTER>**.
G = records by group name: When this option is chosen the following menu is displayed:

Compute statistics by group. A = All groups
S= Start/stop groups
G= Select groups

A = All groups: This option allows groups that are labeled to be processed together.

S = Start/stop groups: This option prompts the user for start and stop group names.

Compute statistics by group.	A = All groups
Enter start group. (W) (W)	S = Start/stop groups
Enter ending group. (T) ( )	G = Select groups

Default names are given which represent the first and last group names listed in the data file. If an incorrect name or **<ENTER>** is given, the default names will be used. All groups between and including the start and stop groups will be processed.

G = Select groups: This option prompts the user for group names to be processed.

Compute statistics by group.	A = All groups
	S = Start/stop groups
Enter 1st group name. ( )	G = Select groups

When the user enters a group name it is checked to assure the name exists in the data file. Up to 99 group names may be entered. Group specifications are ended by responding with **<ENTER>**.

Menu options: When computations are complete or when a statistical analysis is reprinted the following menu is displayed:

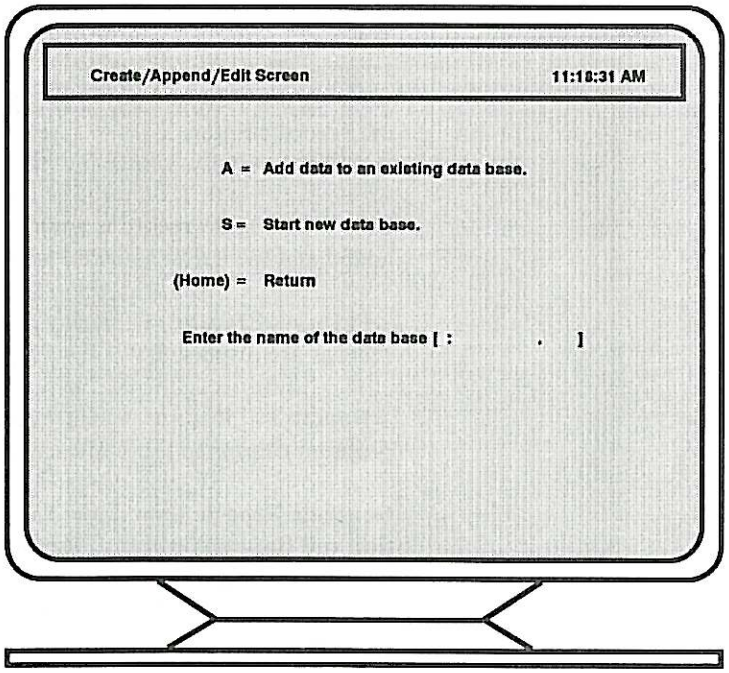
Statistical Analysis Complete	S = List
	P = Print
	B = Bargraph
	<Home> = Return

S = List: When 'S' is chosen, all records are displayed by pages. This is a quick way to review all the records in the statistics file.

P = Print: When 'P' is chosen, a preliminary report is generated. This report is useful as a quick check or for in-house use.

B = Bargraph: See page XXfor a full explanation of the bargraph generator.

<Home> = Return: When **<Home>** is chosen the program returns to the View/Label/ Compute screen if the data file was previously displayed, or the Main screen if the data file was not previously displayed.



Create/Append/ Edit Overview: When this option is chosen from the main screen this screen is displayed:

This part of the program is used to manually enter information in a data file. The user may choose to create a new data file or use an existing one. Data already entered or collected by DATATRAP and processed into a data file may be modified. The primary purpose of this function is to create a data file which can be used to create a statistics file.

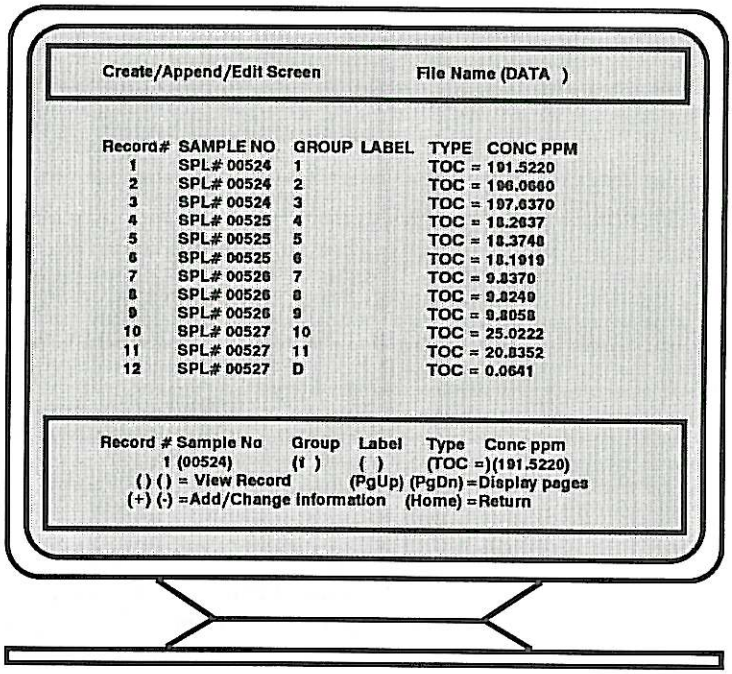
Create/edit files: The data file needs to be located in the main or currently selected directory.

A = Add data to an existing data base: The user is asked for a drive letter, file name and file extension. If a drive letter is not specified drive A is selected. If an extension is not entered the file will be given the extension of '.DBF'.

S = Start new data base: This option operates in the same manner as above.

<Home> = Return: This will return the user to the Main menu.

Menu/display: After a file name is accepted the following screen is displayed:



< >< > = View Record: When the up or down arrow is selected, one record at a time is displayed inside the menu area. This is the process used to edit an existing record.

<-> = Edit record: When the <-> key is used, the displayed record is available for editing.

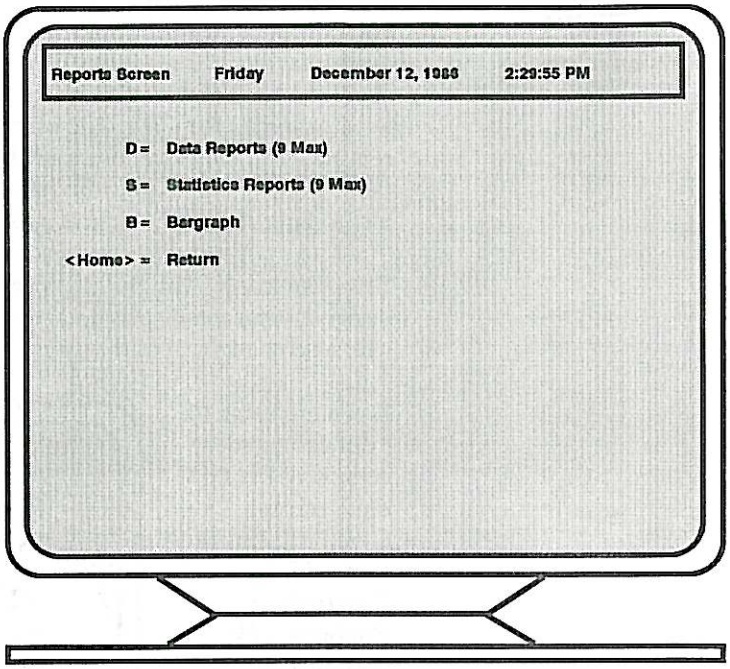
<+> = Add record: When the <+> key is used, the record pointer is positioned to the end of the file where a new record can be added. The menu area will display blank fields to be filled in. If information is left out the bell is sounded once and the new record is added. If all fields are left blank a new record is not added and the bell is sounded twice.

<PgUp><PgDn> = Display pages: When these keys are selected pages of twelve records are displayed.

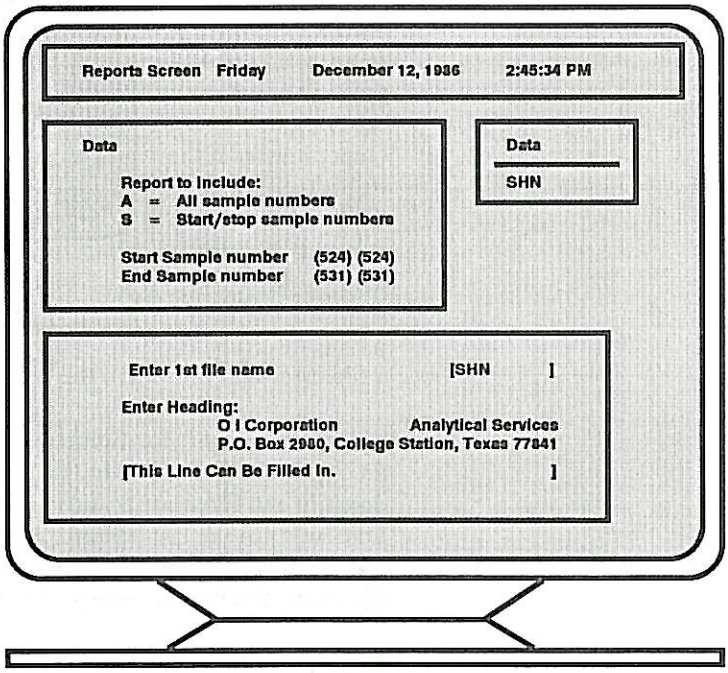
<Home> = Return: When <Home> is selected the data file is saved and the program returns to the Main screen.

Generate Reports Overview: This section of the DHS is used to create Data, Statistics, and Bargraph reports. Reports generated here include filename, date, and blanks to be filled in by the operator. The Data report shows samples from the data (.DBF) file. This report may be restricted to a limited number of samples. The Statistics report uses the statistics (.STA) file. The DHS will determine how the

statistics file was computed and allow the user to selectively limit the information that will go into the Statistics report. When creating Data or Statistics reports, the user is allowed to queue up to nine reports before starting the printing process. These reports may be created from the same file. The Bargraph Generator will produce one custom report at a time. This report may be based on a data or a statistics file. When Generate Reports is chosen from the main screen this screen is presented:



D = Data Reports: When this option is chosen, the following screen is presented:



The user is required to enter a filename without a drive letter or an extension. If this file exists in the currently selected directory, the filename is put in the queue located at the right side of the screen. The company header is displayed with an optional blank line. The line will not appear in the report if the user responds with **<ENTER>**. The program then prompts the user for a limiting factor.

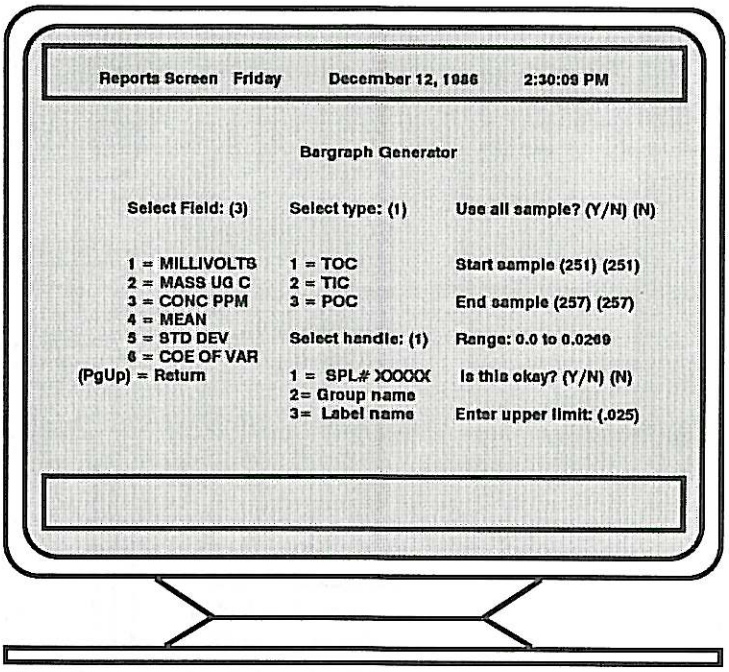
A = All samples: When this option is chosen, the report includes all active samples in the data file.

S = Start/stop sample numbers: This option allows the user to limit the report to a select set of samples. The user is prompted for start and stop sample numbers. If incorrect responses are given or **<ENTER>** is used, the report will use the default numbers provided.

G = Group: When this option is chosen, the user is prompted for a group name. This function produces a report consisting of samples from the designated group. The user will be reprompted if the name entered is not found in the data file. If no group name is found, the user can respond with <enter> and the report will include all samples.

S = Statistics Reports: The procedure for Statistics reports is the same as for Data reports. The only variation is in chosing a limiting factor. After a filename is entered, the DHS determines how the statistics were computed. The user is given the opportunity to limit the report by group, date, or sample number.

B = Bargraph: The Bargraph generator presents the user with prompts used to design a bargraph display and report. The prompts are displayed in succession ending with a screen typical of that presented below:



If you are using the extended memory version of the DHS the Bargraph Generator can be accesed from the **View/Label/ Compute** and the **Statistics** screens. If the 256K version is used the only access is from the reports screen. In the case of entering the Bargraph Generator from the Reports Screen a filename is requested. If a drive letter is not supplied, the DHS path is inserted. If a file extension is omitted the filename will be given the extension of .DBF.

The first prompt is for 'Select Field.' The user is presented with six possible fields and an opportunity to return to the previous screen. Three of these fields are from the data file and three are fron the statistics file. The user may choose from any field. The DHS will use the last filename entered and attempt to locate and read in the correct file. If the field contains all zeros, the user is prompted for another selection. If the file exists, the user is prompted for 'Select type' which will be TOC, TIC, or POC.

The user will then be asked for a handle. This is used to reference the lines of the display and the report.

The next prompt allows limiting the sample range. If the user answers 'N' (No) to the 'Use all samples?' prompt, starting and ending default numbers are displayed.

The Bargraph Generator includes an auto-ranging display. The range presented is for the largest value found within the previously set limits (field, type, sample range). If the value presented is not desired, a different value may be entered. The screen and report will show a '+' at the end of the bars that exceed the range. After completing this screen, the following menu will be displayed:

Bargraph
P = Print
<Home> = Return
D = Display next page
R = Reconfigure Generator

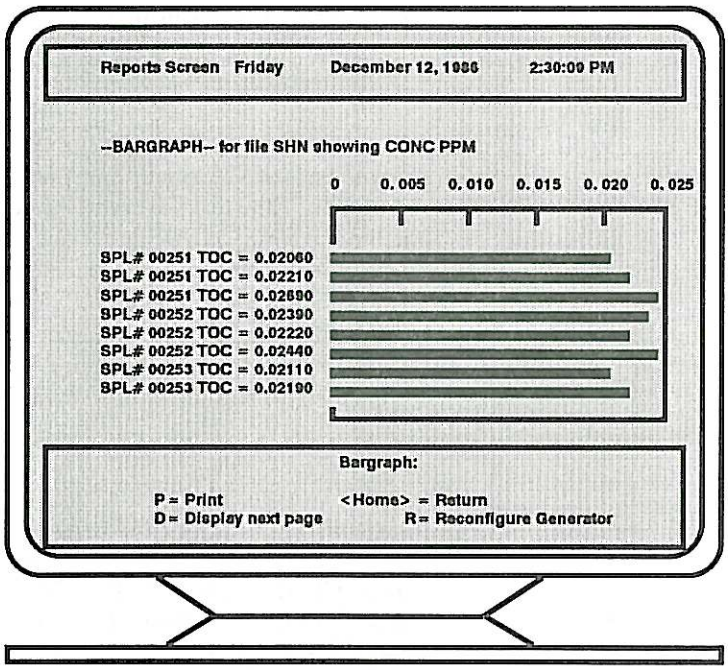
P = Print: When this option is chosen, a bargraph will be printed.

D = Display next page: This option allows the user to view the bargraph on the screen one page at a time.

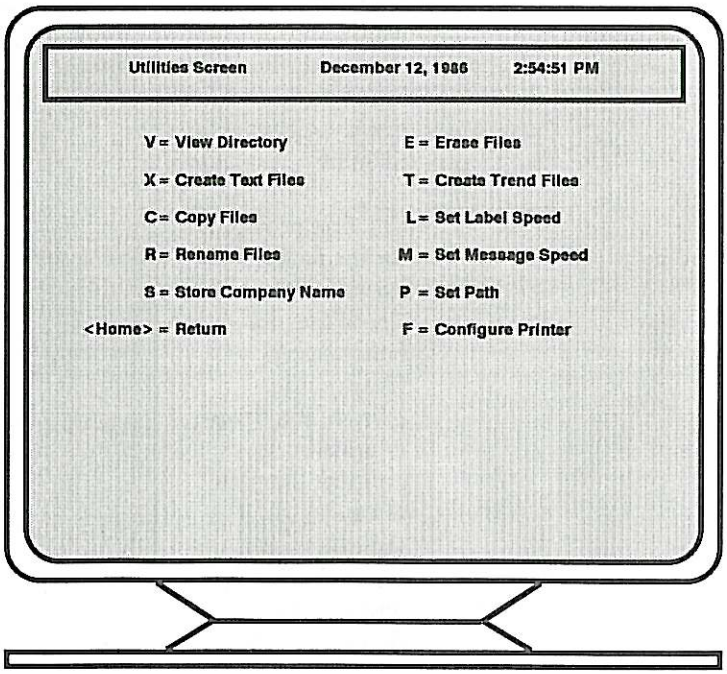
R = Reconfigure generator: This option re-displays the Bargraph Generator and allows the user to change previously set parameters.

<Home> = Return: When this option is chosen, the user is returned to the previous screen.

This screen is an example of a bargraph display:



Start/Stop Data Collection: This screen shows the commands associated with the background data collection program DATATRAP. After DATATRAP has been initialized, the user may change the any of the parameters by starting a new data file from this screen. If the change was implemented DATATRAP will respond with 'DATATRAP changed to new parameters'.



Utilities Screen Overview: The Utilities screen gives the user the ability to modify certain DHS parameters and process common DOS operations. In all cases a complete filespec may be entered. If a drive desiginator is omitted, the

current path will be used. Although DOS global symbols may be used this can cause screen conflicts on some computers. If massive file manipulation is required it may be better to do these from the DOS prompt or with a file program. The previous screen is presented:

DOS Commands

V = View directory: When this function is chosen, the user is asked for a drive letter and is given the opportunity to limit the files to be displayed.

C = Copy files: This function gives the user the opportunity to copy a file and rename it in the process.

R = Rename files: This function gives the user the opportunity to rename a file.

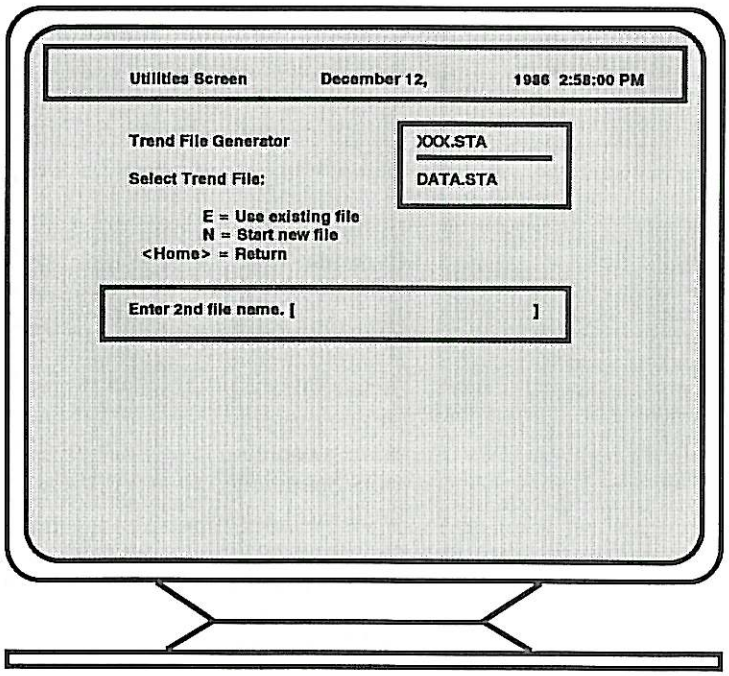
E = Erase files: This function will erase (delete) a file.

DHS functions include the following:

X = Create Text files: This option allows the user to take a Data or Statistics file and create an ASCII formatted text file. The text file can be read by other PC compatible programs. The text file created can be in Space (SDF) or Comma (DELIMITED) delimited form. If SDF is chosen, refer to Table 1 for the database structure. After typing 'X' a filename prompt for the name of a database is presented. If the drive is not given, the path will be used. If an extension is omitted, a .DBF will be appended. If this file exists the user will be asked for the name of the text file that will be created. If the drive is omitted, the path is used. If the extension is omitted, a .TXT will be appended. If this file already exists, the user will not be allowed to overwrite it.

S = Store company name: This function gives the user the opportunity to to enter one or two lines that will become the header on the Data and Statistics reports generated at the Reports Screen.

T = Create Trend file: This option gives the user the ability to join up to nine statistics files to form one file. This feature is most useful when statistics were computed by date and many days activity need to be displayed. The following screen is displayed and filled in:



The first prompt is for the name of an existing or a new Trend file. In either case a filespec is required. If a drive desiginator is omitted, the path will be used. If the extension is omitted, .STA is added to the filename. When

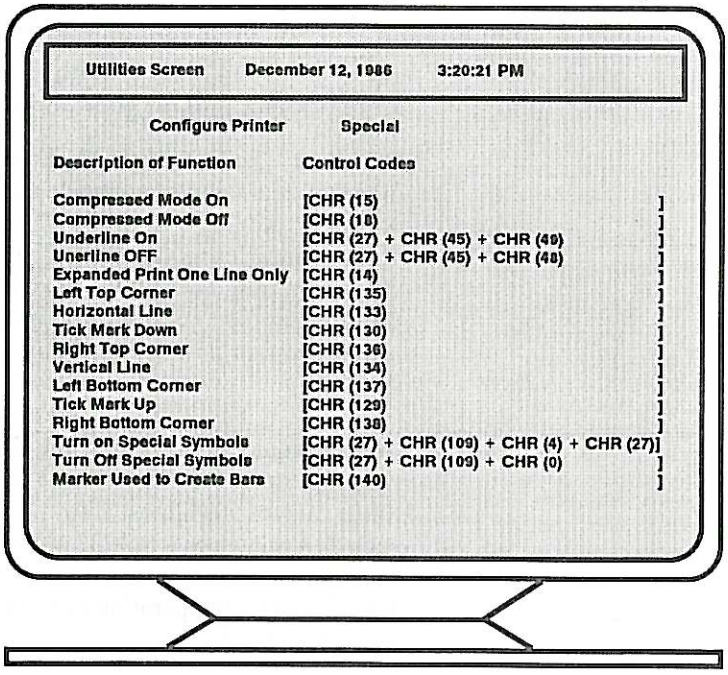
the DHS creates or locates the Trend file, the filename is displayed and a queue of files is shown along the right hand side of the screen. The files to be concatenated must be located in the current directory. To end filename entry, use the **<PgUp>** or **<ENTER>** key. As the files are joined, an arrow will be displayed next to the filename in the queue.

L = Set Label speed: This function allows the user to select a number between 0 and 999. This number controls the speed of the 'L = Label samples' function on the **View/Label/Compute** screen. The normal speed is 200 for a PC compatible computer.

P = Set Path: This option allows the user to set a search path other than the current directory. This path is used where a drive letter is omitted. The path must end with a backslash (for example C:\mydata\).

M = Set Message speed: This function gives the user the ability to control how long an error message will be displayed. The user may enter a number between 0 and 999 with 200 being typical.

F = Configure Printer: When this function is chosen, the user is able to configure the DHS to print properly with the Epson RX-80, Okidata 182, or a printer which is compatible to either. Presented here also is a screen to change print commands used to control the printer. This screen can be used to change the symbols used for the Epson and Okidata printers or to enter commands for a printer not in the configure list. If 'S' is chosen, the following screen is displayed:



The commands presented on this screen will be those of the last printer type selected. All commands must be in the CHR ( ) form shown.

<Home> = Return: When this key is selected the user is returned to the Main Screen.

Exit to DOS: When entering '0' the program terminates and control is returned to DOS.

Control Keys

<Home> ..... Moves the cursor to the beginning of the current blank.

<Ctrl><Home> ..... Moves the cursor to the beginning of the first blank.

<End> ..... Moves the cursor to the end of the current blank.

<Ctrl><End> ..... Moves the cursor to the beginning of the last blank.

Up arrow	Moves the cursor to the previous blank.
Down arrow	Moves the cursor to the next blank.
Right arrowMoves.....	Moves the cursor one character to the right.
<Ctrl> Right arrow	Moves the cursor to the beginning of the next word.
Left arrow.....	Moves the cursor one character to the left.
<Ctrl> Left arrow	Moves the cursor to the beginning of the current word.
<Ctrl> T	Deletes the word to the right of the cursor.
<Ctrl> Y	Deletes the rest of the field to the right of the cursor.
<Alt> C.....	Terminates entry if in a blank or is emergency escape if not in a blank. Always try to use 'Q' in response to the prompt. If 'Q' does not work use 'A'.
<PgUp><PgDn>	Used to exit a blank with out an entry.
Esc.....	Terminates entry without saving it's value.
<Ctrl>	Restores the current blank to it's original value.

Process Sample Multiplexer

Overview: The OI Sample Multiplexer is designed to allow unattended sequential monitoring of up to four sample streams for TOC analysis by the Model 700 TOC Analyzer. Additional streams (up to 8) may be monitored by combining or stacking sample multiplexers. Analysis is performed by activating one of the dual three way valves, diverting normal flow into the sample loop of the TOC Analyzer. At the end of the analysis, the next valve is activated, and the cycle is repeated as the Model 700 microprocessor advances the sequence. Replicate samples may be programed if desired.

System requirements include the Model 700 with Process Sampling option and a means of sampling the streams of interest (i.e. plumbing up or down to 1/8" tube). Since analysis will almost always be performed unattended, a data handling system such as a printer or a RS-232 computer link will be necessary. If high and/or low TIC or TOC concentrations are to be screened, alarms for these functions are also needed. Descriptions and operation of these required components are given in **Chapters 2** and 3 of this manual.

Description of Components

Controls: Consists of a RESET button that sets sequence back to beginning, a STEP button which allows user to manually change channels, and four numbered LED's showing valve actuation.

Electrical - External: Consists of a dual AC plug assembly, the lower plug for a conventional three wire power cord, the upper plug terminal supplies AC power to a second optional Multiplex Sampler via a male-female plug-type power cord.

A fuse compartment for GMA-1 type fuses is located below the lower plug. External control is supplied via the two wire terminal block to the left of the AC plug assembly.

Plumbing - External: Up to four sample streams may be plumbed to the multiplex sampler through the 1/8" TFE INLET lines labeled 1-4. Waste sample is returned through the lines marked OUTLET. The PRIMARY OUT line routes the sample to be analyzed to the Model 700 TOC Analyzer. If two Multiplex Samplers are to be connected in series, the connection is made through the port marked Primary In.

Plumbing - Internal: Consists of up to four, dual three way TFE valves used to sequentially route different samples to the Model 700 TOC analyzer. Incoming sample lines are coded with blue fittings. Drain or waste lines are coded green. Valve to valve lines are coded red. Sample loop connections are coded with clear fittings. All tubing and connectors are 1/8" x .063" teflon and 1/4"-28 x 1/8" fittings, respectively.

Electrical - Internal: Consists of a Sample Stream Multiplex Control board. The board has a terminal strip for connection of the four switching valves, along with panel mount selector toggle switches and panel mount DIP switches for valve programming.

Installation

General Information: The Multiplex Sampler is designed to operate along side the Model 700 TOC Analyzer. Connecting plumbing supplied with the sampler includes four feet of teflon tubing to be attached to the Model 700 internal plumbing. The sample inlet lines are also four feet in length. As the samples to be analyzed have positive pressure, the module need not be at the same level as the Model 700.

Removing Module Cover: Internal components may be inspected by removing the two shipping screws located at the bottoms of the right and left side panels. After removing screws, slide top panel to rear approximately 1/4" and lift up. It is recommended that the cover be in place during normal operation.

Installing Multiplex Sampler: Upon opening shipping box, inspect for complete shipment. Included should be the Sampler Module, power cord, and external control cable.

- Locate the sample module beside the Model 700. Check to see that the power cord will reach an external 110 VAC line or the accessory power plug at the rear of the 700.

- Remove the left bay cover from the Model 700. Disconnect the process sampling port line at the coupling Port 4 of Valve C (**Chapter 7**). Connect "PRIMARY OUT" line from sampler to this coupling. Remove pump cartridge retaining bail from pump head and see that the pump cartridge is free from the pump rollers.

- Plumb sample inlet lines 1-4 as needed to the streams to be monitored. Inlet sample pressures should be adjusted to less than 75 psi. Sample should begin to flow from the outlet lines. Plumb outlets to waste as desired.

- Attach the two wires of the control cable to the rear of the Sampler Module at the two-position terminal strip. As viewed from the rear, the black wire is on the left, red on the right. Plug the other end of the cable into the connector marked "EXTERNAL CONTROL" at the rear of the Model 700.

- Attach power cord to the bottom plug of the dual plug connector at the rear of the Sampler module. Plug cord into 110 VAC source. Look to see that one of the four LEDs lights up on the front panel. Verify that the STEP button actuates the respective valves and that liquid flows through the sample loop of the Model 700.

Operation

Power Up: Remove cover from Sampler Module. Check settings of the circuit board mounted toggle switches (set of two) and DIP switches (set of eight).

Dip Switches: The series of DIP switches, numbered 1-8, selects the total number of streams to be analyzed. Set all switchs off (down/open) except for the last valve position be be sampled. To sample three streams, for example, set all switches down except for switch #3.

Toggle Switches: The toggle switches designate the sampler as #1 (valves 1-4) or #2 (valves 5-8), if two samplers are coupled together for analysis of more than four streams. The toggle switches would be placed up on a second Sampler Module. For normal, four stream analysis, both toggles are in the down position.

- Replace Sampler cover and plug into 110 VAC power. Push the RESET button to set valve sequence to 1.

- Startup and calibration of the Model 700 is described in Chapter 4 of this manual. Adjust reagent and sample volumes as needed.

- Review Model 700 system configuration. Sections 1 and 2 of the Set System Configuration menu should be set as follows:

Acid Pump	- enabled	Autosampler	- enabled
Oxidant Pump	- enabled	Ready/Stand	- disabled
Sample Valve	- enabled	Sample Number	- as desired
Sample Pump	- disabled	# Reps	- as desired
Auto Run	- enabled	Sample Stop	- as desired
Auto Print	- enabled	Alarms	- as desired

NOTE: Do not overtighten the fittings that connect with the TFE solenoid valves. If a fitting will not seal after finger-tightening, examine tubing flare and re-flare if necessary.

- Observe that sample flows through the drain lines to waste. If bubbles are present, check for loose fittings along the suspect flow path. Place the 700 sample loop in-line with a sample stream and check for bubbles. Check fittings as needed.

- Press the "Clear" button on the Model 700 and allow system to drain for 10 seconds. Press Run/Stop button to begin analysis.

Maintenance Definitions

1/8" TFE tubing - OI #147901, 1/8" OD x .062" ID teflon tubing.

1/8" tube fittings - stocked in several colors. Includes washer.

Green	OI #166357
Blue	OI #166381
Red	OI #166365
Natural	OI #165862

These are nylon or polypropylene fittings with 1/4"-28 thread and a 5/16" head.

Coupling - OI #166274 - a nylon or polypropylene fitting with internal 1/4"-28 threads for connecting two tube fittings.

Leak Check: Periodically remove the top cover from the Sampling Module and inspect for liquid leaks. Contact with some solvents may degrade the tube fittings in time. If necessary, replace with a more inert material (fluorcarbon or a more resistant plastic).

Flow Obstruction: Inspect drain lines to see that adequate flow is present. For sample streams with high particulate loads, an in-line filter may be necessary. Remove any valve obstruction by backflushing with a syringe.

Chapter 9
Plumbing Schematics

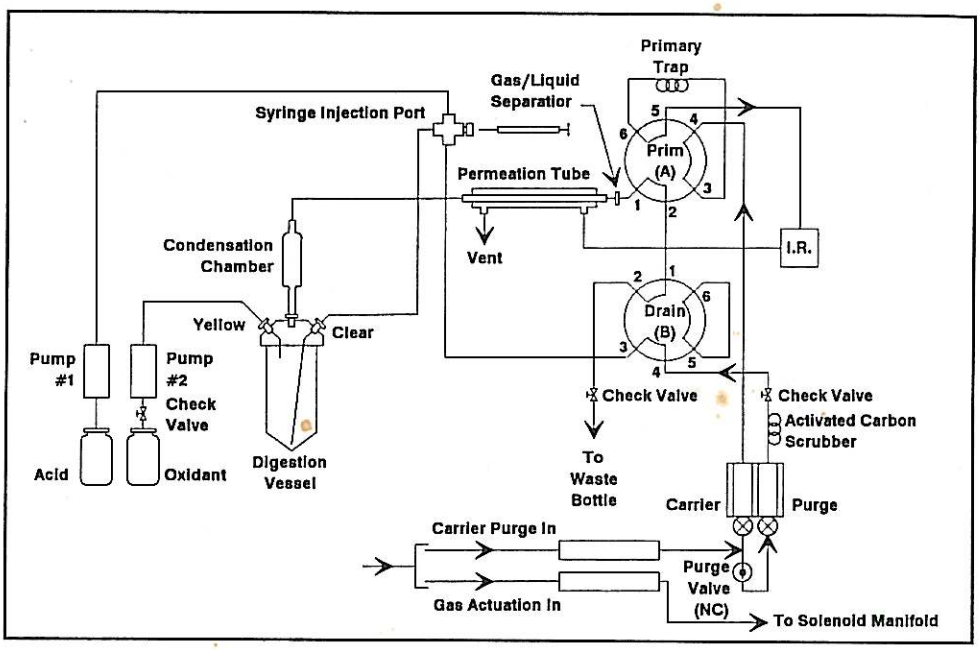


Fig. 9.1 Basic Unit

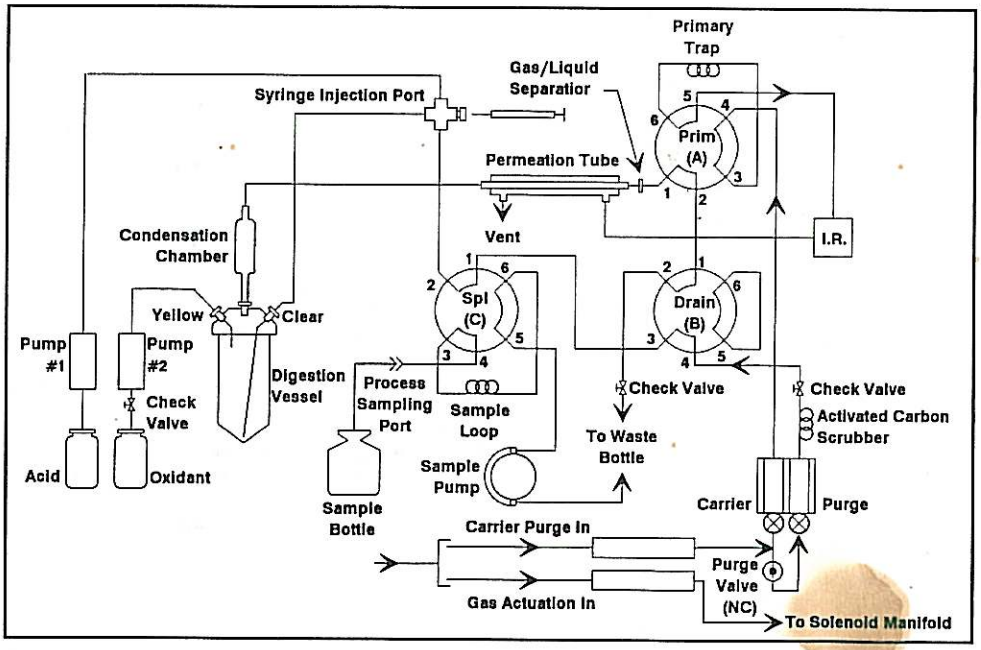


Fig. 9.2 Basic Unit w/Process Sampling Option

Plumbing Schematics (cont.)

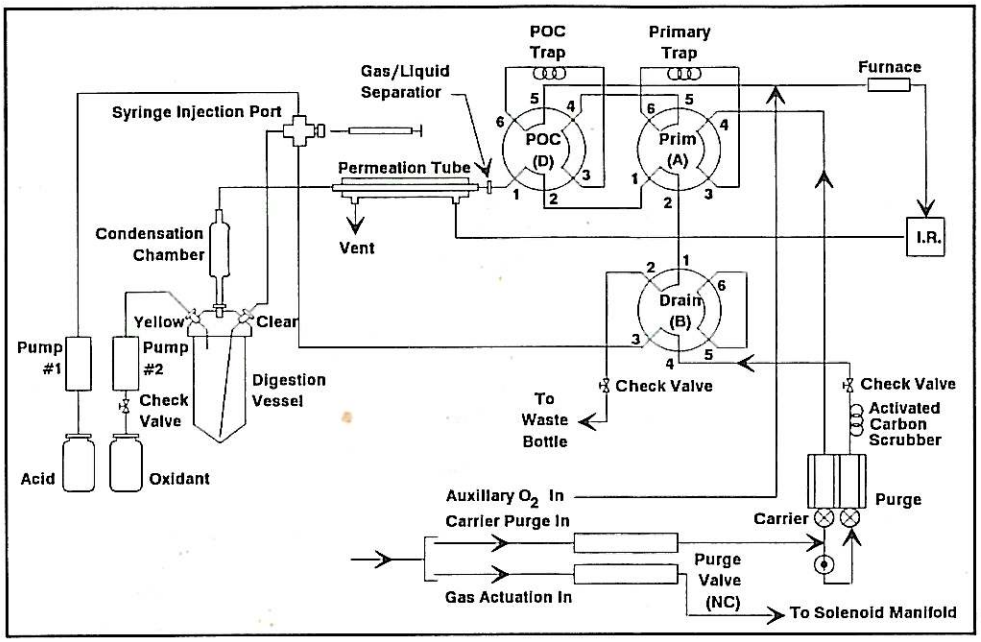


Fig. 9.3 Basic Unit w/Purgeables Option

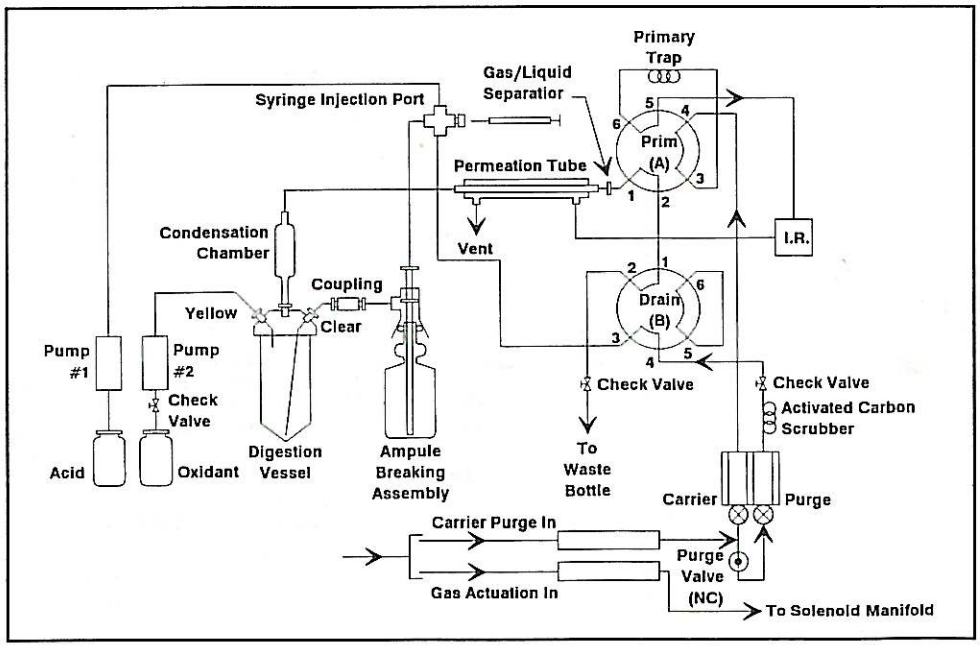


Fig. 9.4 Basic Unit w/Ampule Option

Plumbing Schematics (cont.)

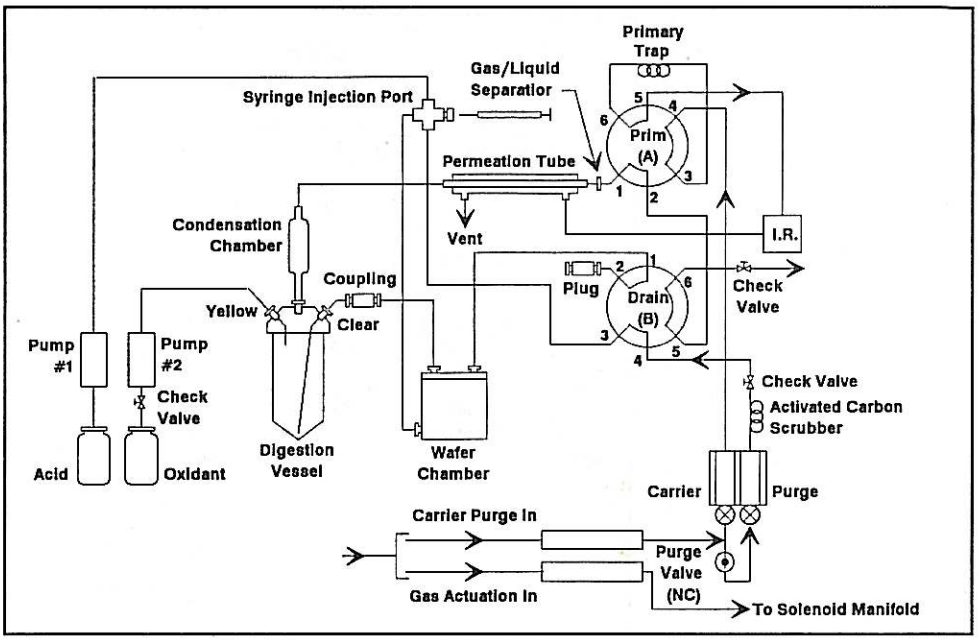


Fig. 9.5 Basic Unit w/Wafertoc Option

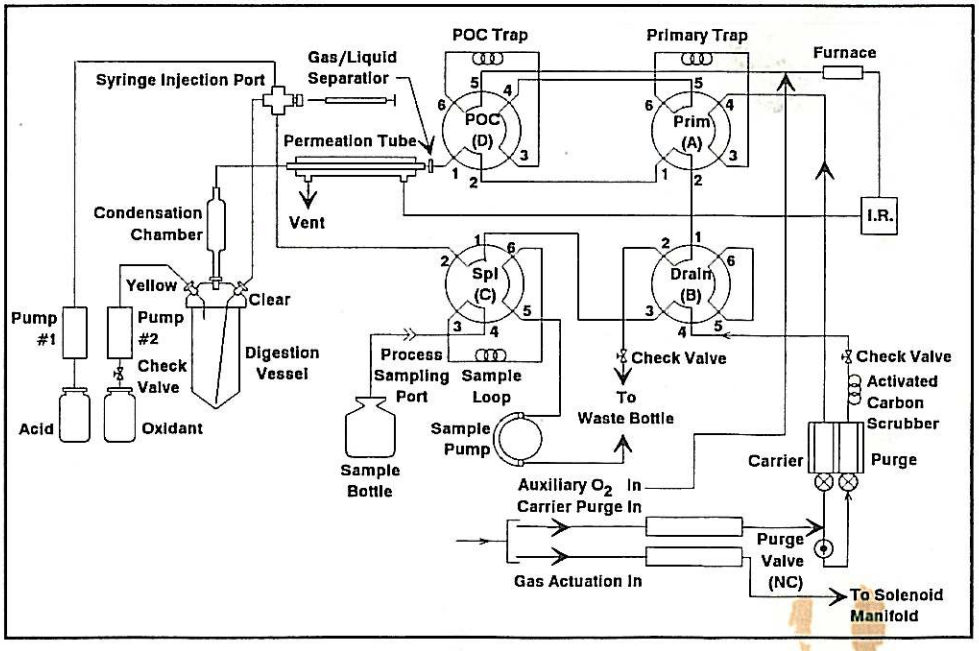


Fig. 9.6 Basic Unit w/Process Sampling & Purgeables Options

Plumbing Schematics (cont.)

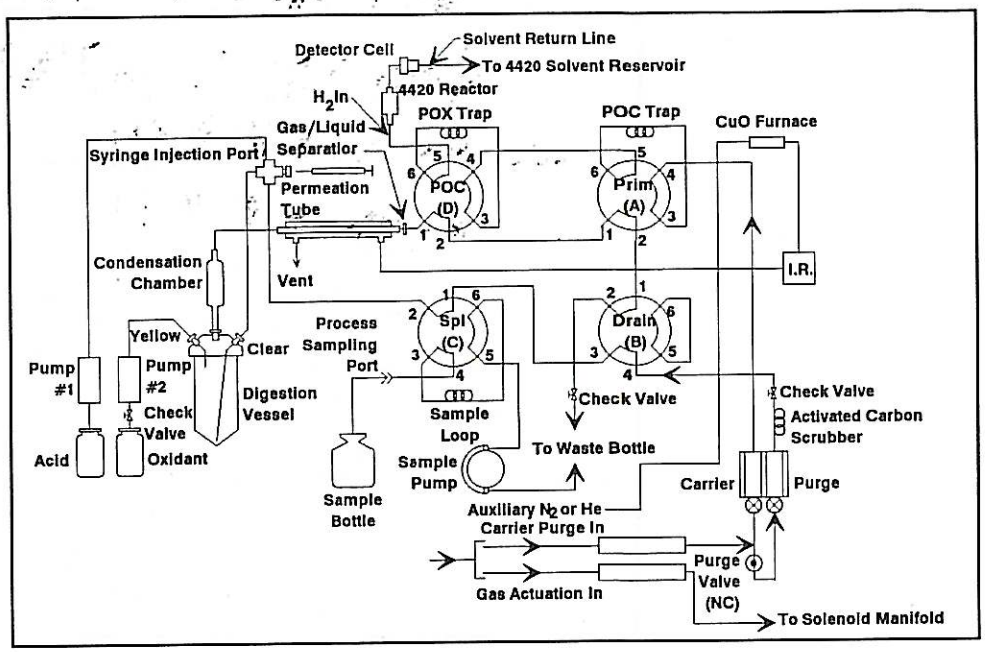


Fig. 9.7 Basic Unit w/Process Sampling & POX Option