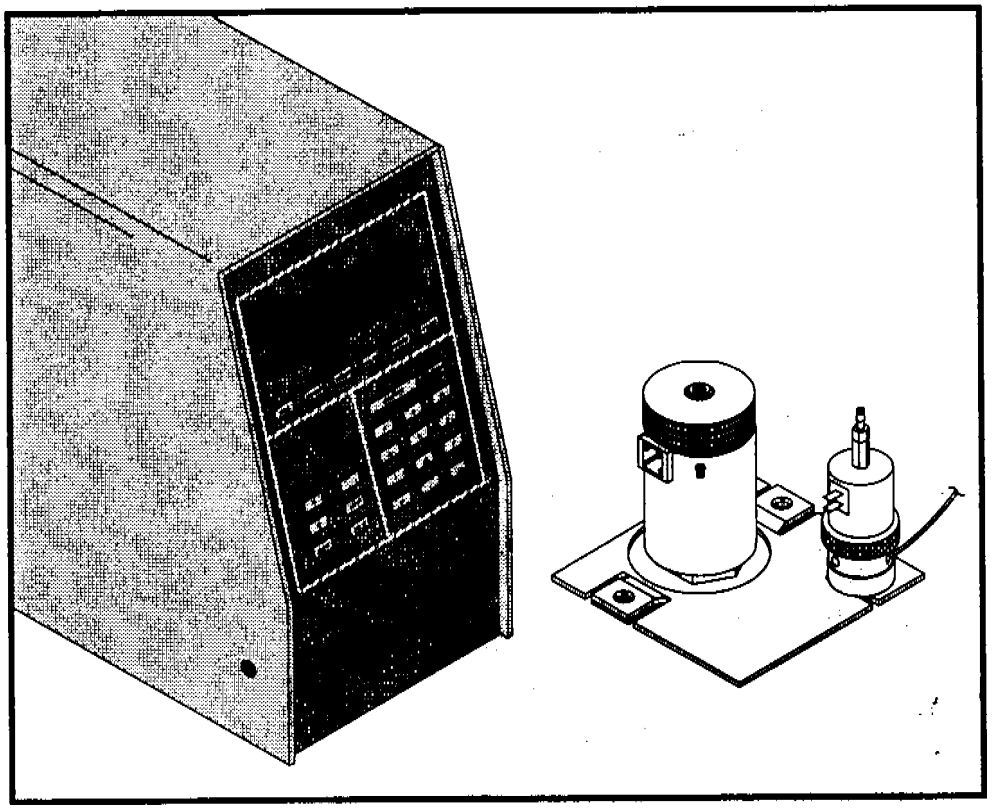
Model 5220 Electrolytic Conductivity Detector Operator's Manual



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Chapter 1
Introduction

The Electrolytic Conductivity Detector (ELCD) offers the widest range of specific compound detection capabilities of any common gas chromatograph (GC) detector. Operating conditions are available for the selective detection of halogen, sulfur, and nitrogen compounds.

OI Analytical's Model 5220 Electrolytic Conductivity Detector represents a true advance in detection technology. The 5220 has been specifically designed for the selective detection of halogens (H), sulfur (S), and nitrogen (N) present in compounds eluting from capillary GC columns. Major improvements have been made in all detector components, including the reactor, the conductivity cell, electronic systems, and fluid-flow systems.

Detector Design

The OI Analytical Model 5220 consists of three principal components (see Fig. 1.1):

- 5200 Detector Controller
- Reactor Assembly
- 5220 Cell/Solvent Assembly

The 5200 Detector Controller houses signal processing, control, and power supply electronics. It is functionally independent of the GC. The 5220 Cell/Solvent System houses the Cell, Conductivity Amplifier, and Solvent System. The Reactor Assembly houses the Reactor, Reactor Base, and Vent Valve.

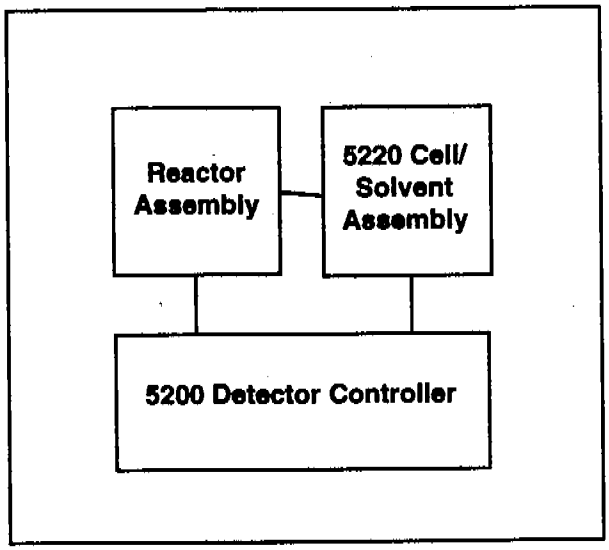


Figure 1.1. Model 5220 ELCD Principal Components

The three main operating modes for the 5220 are: Halogen (H), Sulfur (S), and Nitrogen (N). OI Analytical offers an operating kit for each of these modes. Each kit contains all of the required materials (except the electrolyte solvent) for operating the Detector in the specified mode.

Principal of Operation

A continuously running solvent pump pulls electrolyte (solvent) from the Solvent Reservoir, then pushes the electrolyte through the Resin Cartridge and Solvent Block into the Detector Cell. A return path from the Cell to the Solvent Reservoir is also provided for the electrolyte. As the effluent from the GC column enters the Reactor Base, it mixes with a reaction gas and flows into the Reactor. The Reactor and Reaction Tube act with the reaction gas to convert species of interest to ionizable gases.

These gases are carried into the Cell Assembly and come in contact with the electrolyte, where the ionizable gas dissolves in the electrolyte and increases the electrolytic conductivity of the mixture. This increase is measured by a pair of Conductivity Electrodes in the Cell Assembly working in conjunction with the Conductivity Amplifier. The change in conductivity is converted to a voltage signal that is processed by the 5200 Detector Controller and then quantified by an external data output device, which results in a chromatogram.

The Vent Valve and Controller provide the option of either venting the injection solvent before it is carried into the Reactor, or venting at another time determined by the analyst during a run. Carbonaceous solvent can cause elemental carbon buildup in the Reactor under certain conditions, interfering with proper response to compounds of interest. Oxygenated solvents may immediately and irreversibly foul the Reaction Tube. Reactor temperature and electrolyte flow rate both affect sensitivity, so the Controller is designed to adjust and optimize their settings.

NOTE: The performance effects caused by solvents are limited to direct injection of organic solvents. Purge-and-trap analysis is usually not affected by these problems. Sufficient amounts of organic solvents are rarely purged from solution.

The principle of response of the 5220 is based on the conversion of the analyte to small, ionizable, inorganic molecules, and on detection of these molecules by a change in electrolytic conductivity after partial dissolution. In this process, there are three primary steps that take place:

- pyrolytic conversion of the analyte to a monitored species;
- partial extraction of the monitored species from the gaseous reaction products stream into the conductivity electrolyte; and
- detection of the monitored species by the change in resistance of the resulting mixture.

These processes occur entirely within the Reactor and Cell Assemblies in a continuous manner. The GC column effluent is mixed with reaction gas (H_2 or air) in the Reactor Base. The mixture then flows through a Reaction Tube within the Reactor where that sample is pyrolyzed at temperatures from 700 to 1100°C. The reaction products are swept into the Cell Assembly where they are mixed with the deionized electrolyte, then the electrical resistance of the resulting solvent-gas mixture is measured.

Selectivity for a given element depends upon:

- the reaction conditions used for converting the analyte to the monitored species;
- the use of chemical scrubbers for removing interferences; and
- the type and pH of the electrolytic conductivity solvent.

Specific operation details for the various operating modes are summarized in Table 1.1.

Depending on the reaction gas used, conditions within the Reaction Tube can be reductive (H_2) or oxidative (air). The various reaction products produced in the three primary operating modes and the basis of selectivity are summarized in Tables 1.2 through 1.4.

Operating Parameters for Selective Detection

Mode	Rx Temp (°C)	Rx Gas	Conductivity Mode Solvent	Scrubber
Halogen (H)	850 - 1100	H_2	100% ACS Reagent Grade n-propanol.	None
Sulfur (S)	800 - 1100	Air	100% ACS Reagent Grade methanol.	S-Mode
Nitrogen (N)	775 - 1100	H_2	90:10 (v/v) 18 megaohm/cm or better deionized, degassed water/ ACS reagent grade t-butyl alcohol.	N-Mode

Table 1.1. Table of Operating Parameters for Selective Detection

Main Reaction Products for X-Mode Detection (Nickel Reaction Tube, Reductive Conditions)

Compound Type	Combustion Product(s)	Selection
Halogen	HX	Selectively detected
Sulfur	H_2S	Poorly ionized
Nitrogen	NH_3	Poorly ionized
Hydrocarbon	CH_4 (lower alkanes)	Not ionized
Oxygen	H_2O	Poorly ionized

Table 1.2. Main Reaction Products for H-Mode Detection

Main Reaction Products for N-Mode Detection (Nickel Reaction Tube, Reductive Conditions)

Compound Type	Combustion Product(s)	Selection
Nitrogen	NH_3	Selectively detected
Halogen	HX	Removed by post-reactor scrubber
Sulfur	H_2S	Removed by post-reactor scrubber
Hydrocarbon	CH_4 (lower alkanes)	Not ionized
Oxygen	H_2O	Poorly ionized

Table 1.3. Main Reaction Products for N-Mode Detection

Main Reaction Products for S-Mode (Alumina Reaction Tube, Oxidative Conditions)

Compound Type	Combustion Product(s)	Selection
Sulfur	SO_2	Selectively detected
Halogen	HX	Removed by post-reactor scrubber
Nitrogen	N_2, NO	Not ionized
Hydrocarbon	CO_2 (lower alkanes)	Poorly ionized
Oxygen	H_2O	Poorly ionized

Table 1.4. Main Reaction Products for S-Mode Detection

Features

- Low maintenance cell with quick-disconnect attachments.
- Compact modular design.
- Directly interfaces to several GC makes and models.
- Patented design allows reaction tubes to be replaced quickly and easily.
- Direct interface to OI Analytical Model 5230 Photoionization Detector without transfer line (using single detector port).
- Operator-interface is simple to use (vacuum-fluorescent display is easy to read from any angle).
- Detector Base is designed for capillary columns.
- Reaction gas serves as makeup gas.
- Packed Column Kit is optional.
- New reactor design eliminates solid graphite ferrules, using brass and graphite/Vespel® (GRP/VSP) ferrules.
- Reactor temperature, Detector vent, and solvent flow are microprocessor-controlled and digitally input.
- Control Module incorporates quick-change, disposable deionizing cartridge and simplified solvent system.

- Unit incorporates power-up self-test, diagnostic sensors and messages, and power failure restart sequence.
- PID or second ELCD can be added to form integral dual-detector with all functions controllable by the 5200 Detector Controller.
- All functions of dual detectors can be programmed to turn ON and/or OFF at pre-set times during each run.
- Run-time is programmable, elapsed/remaining time is displayed, and a separate stopwatch is built-in.
- Up to 14 sets of run-settings can be saved and loaded as files.
- Vent(s) may be turned ON and/or OFF on multiple occasions at preset times during each run.

Principal Applications

- EPA 601
- EPA 608
- EPA 611
- EPA 502.1
- EPA 502.2
- Petroleum Products
- Pesticides, HX, N, S
- PCBs
- Pharmaceuticals
- Industrial Chemicals
- Nitrosamines
- Forensic Science

Specifications

Any halogen, nitrogen, or sulfur in a compound eluting from a GC column is converted under reductive or oxidative conditions to an ionizable gas (HX, NH_3, SO_2) in a high temperature catalytic micro-reactor. Gaseous reaction products are carried into a detector cell where they are dissolved with a deionized electrolyte, which increases the electrolytic conductivity of the mixture. This instantaneous change in conductivity is amplified, producing a signal proportional to the mass of halogen, nitrogen, or sulfur in the original compound. Specificity results from the choice of electrolyte, reactor conditions, and scrubber employed.

Modes of Operation

- Halogen
- Sulfur
- Nitrogen

Solvent Vent Valve

- Multiple venting capability, timing in 0.01 minute increments
- Manual or remote controlled

Dimensions (5200 Detector Controller)

- 8.25" H x 5.125" W x 11.5" D

Weight

- 8 lbs.

Dynamic Range

- Halogen 5×10^6
- Nitrogen 0.5×10^6
- Sulfur 1×10^6

Selectivity

• Halogen	Cl/HC >	10^6
	Cl/N >	10^5
	Cl/S >	10^5
• Nitrogen	N/HC >	10^6
	N/Cl >	10^4
	N/S >	10^4
• Sulfur	S/HC >	10^5
	S/Cl >	10^5
	S/N >	10^5

Reactor Temperature

- Range: 0 to 1100°C (direct readout in 1°C increments with ON/OFF control)
- Stability: ±1°C

Solvent Flow

- Operator selectable 10-100% in 1% increments
- Flow Range: 0-100 µl/min

Detector Output

- 1V full scale analog voltage

Gas Requirements

- Halogen Mode: hydrogen, ultra-high purity, 99.999% or better
- Sulfur Mode: air, ultra-high purity, dry, 0.1 molar ppm HC or better
- Nitrogen Mode: hydrogen, ultra-high purity, 99.999% or better

Power Requirements

- 110 VAC (±10%)/60 Hz 2.0A or
- 220 VAC (±10%)/50 Hz 1.0A (switch selectable)

NOTE: Performance is affected by several factors, including GC, column, electrolyte, and compound class.

NOTES

Chapter 2
Description of Components

Principal Components

5200 Detector Controller provides microprocessor control for various components of the Detector as well as conversion of the signal from the Conductivity Cell to a usable output. The 5200 can control up to two Detectors: two ELCDs, two PIDs, or one ELCD and one PID. Electrical components include the Vent Timer, Reactor Temperature Controller, Solvent Pump Controller, Signal Processor, Keyboard, and Display.

5220 Cell/Solvent Assembly includes the Conductivity Cell, its enclosure and mounting hardware, its associated fluid-flow and electrical lines, the Solvent Pump, and the Conductivity Amplifier. This Assembly houses the "sensor" portion of the Detector — the Conductivity Cell.

5220 Reactor Assembly includes the Reactor, Reactor Base, and Vent Valve. A Reaction Tube is installed inside the Reactor. The Reactor Assembly is installed in a GC detector port and accepts a Column in its Base. The GC analytes are converted to ionizable molecules inside the Reaction Tube.

5200 Detector Controller - Front View

Primary Keys are SIG, ZERO, TEMP, VALUE, RATE, TIME, DET, and FILE. Remaining keys (#s, A, B, ON, OFF, ENTER, CLEAR) are used to input information. For specific key operation, see Chapter 4, "Operation."

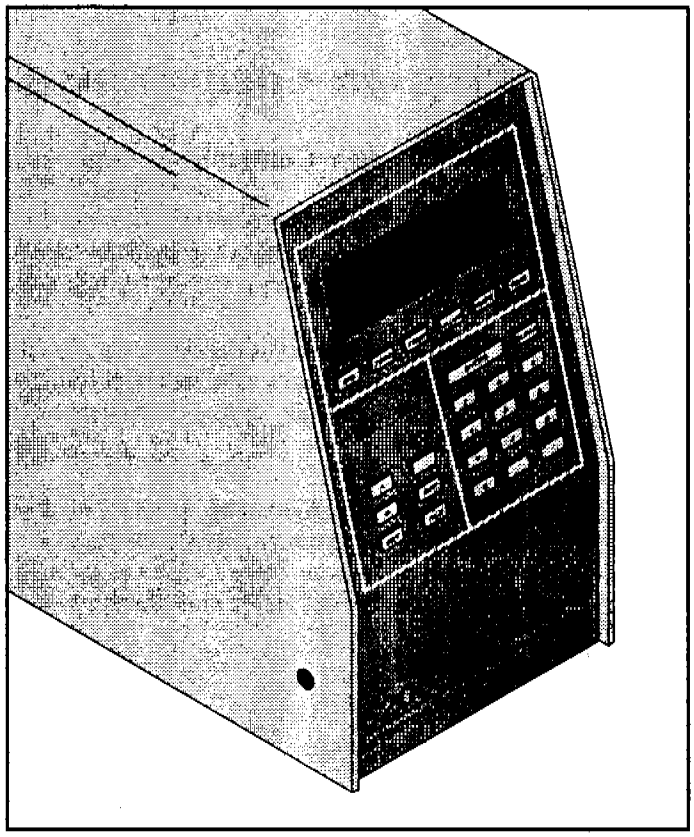


Figure 2.1. Control Module - Front View

SIG is used to view or turn ON/OFF the signal output of Detector A or B.

ZERO is used to zero the signal of Detector A or B.

TEMP is used to set and view Reactor temperature of Reactor A or B. Temperature can be set from 0 to 1100° C.

VALVE is used to view and set ON/OFF time(s) for Vent Value A or B.

RATE is used to set solvent flow rate/percentage and turn the solvent flow ON/OFF. If used with the PID option, RATE is used to set Lamp intensity.

TIME is used to display time elapsed, time remaining, or the stopwatch. It is also used in conjunction with other primary controls to set ON/OFF times.

DET is used to view type of Detector and to turn Detectors ON/OFF.

FILE is used to save, retrieve, and delete Detector operating parameters controlled by the 5200.

5200 Detector Controller - Back View

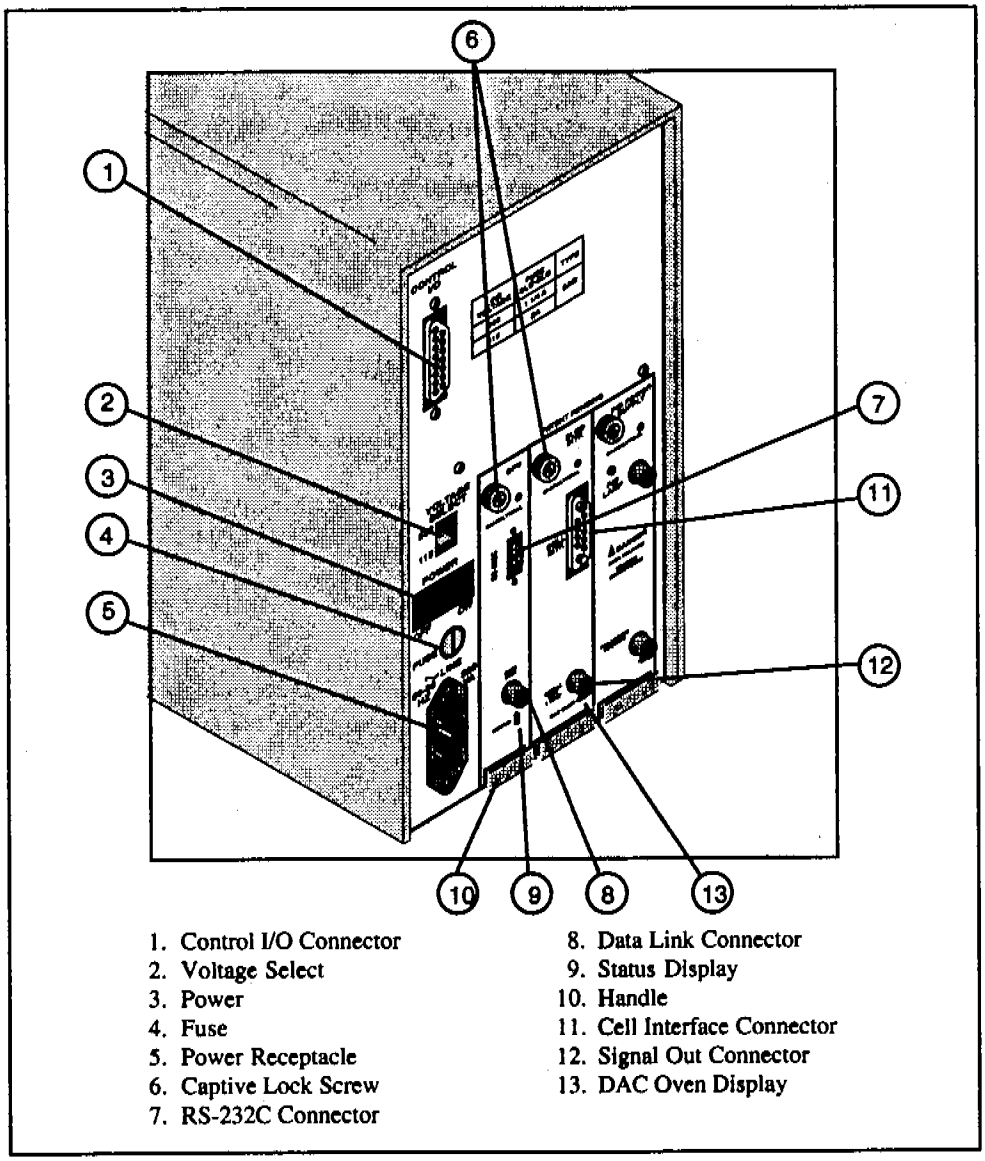


Figure 2.2. Detector Controller - Rear View

Control I/O accepts a contact closure (or 12V or 5V TTL active low) to automatically start the 5200 from an external source.

Voltage Select is set to 110 VAC (±10%) for normal operations (standard outlet voltage). It can be changed to 220 VAC (±10%) as needed.

Power (rocker switch) turns Module ON/OFF.

WARNING:
This receptacle is to be used with a power cord and power source each having a protective earth ground.

Fuse 2 amp slow-blow (110 VAC) or 1.25 amp slow-blow (220 VAC) protects equipment in the event of an internal fault. It can be replaced as needed.

Power Receptacle is an IEC (International Electrotechnical Convention) type power inlet receptacle.

Central Processing Unit (CPU) Board

Captive Lock Screw (spring-loaded thumb screw) is used for locking the Board into place.

RS-232-C Connector allows for standard RS-232-C communications.

Data Link is reserved for future use.

Handle is used to remove the CPU board. The Board should not be removed by pulling on the Captive Lock Screw.

ELCD Interface Board

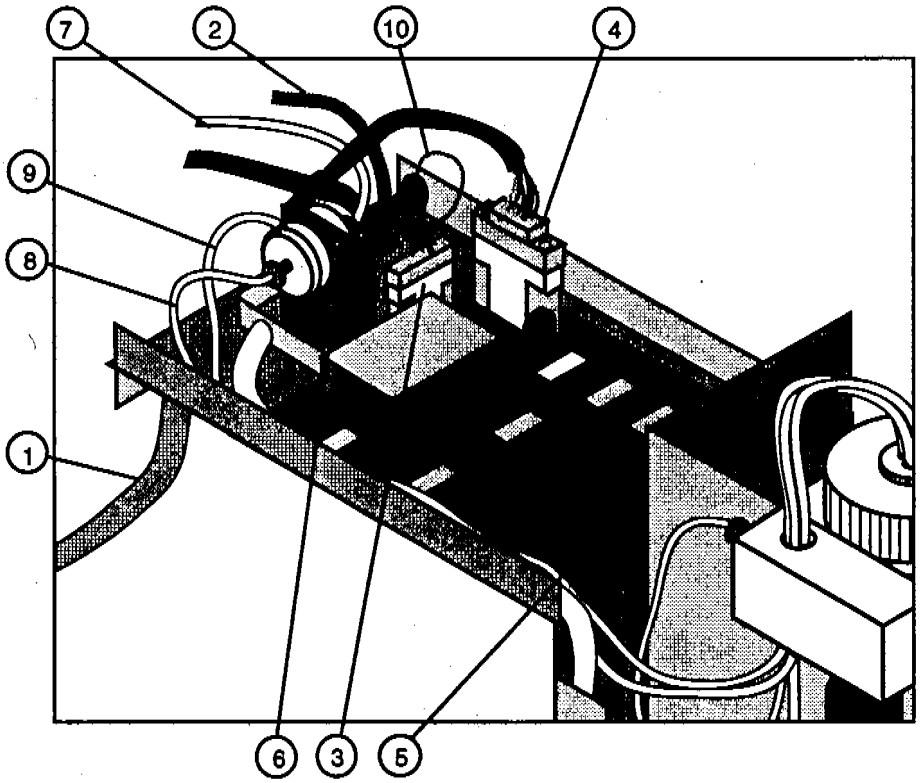
Captive Lock Screw (spring-loaded thumb screw) locks the Board into place.

Cell Interface is a 9-pin connector providing Reactor, Cell, Valve, and Relay closure contacts. The Cell Interface Cable to the ELCD Cell/Solvent Assembly connects here.

Signal Out is a BNC signal connector for 0—1 Volt analog output to a data-recording device.

NOTE: The 5200 may contain 1 or 2 ELCD Interface Boards, 1 or 2 PID Power Supply Boards, or 1 ELCD Interface and 1 PID Power Supply Board.

5220 Cell/Solvent Assembly - Top View



1. Chassis Ground
2. Vent Connector
3. Reactor Connector
4. Cell Interface Connector
5. Pump Connector
6. Cell Connector
7. Transfer Line
8. Solvent Return Line
9. Solvent Cell Inlet Line
10. Assembly Ground

Figure 2.3. Cell/Solvent Assembly - Top View

Chassis Ground is the ground connection to GC.

Vent Connector (2-pin connector) joins with the Vent Valve Cable and makes the connection between the Vent Valve and the Cell Amplifier Board.

Reactor Connector (4-pin connector) joins with the Reactor Interface Cable and makes the power connection between the Cell Amplifier Board and the Reactor Heating Filament.

Cell Interface Connector (9-pin connector) joins with the Cell Interface Cable, interfacing all signal, control, and power lines between the 5200 Detector Controller and 5220 Cell/Solvent Assembly.

Pump Connector (2-pin connector) is for Pump power-out.

Cell Connector (2-pronged connector on U-bracket) allows easy installation of Cell.

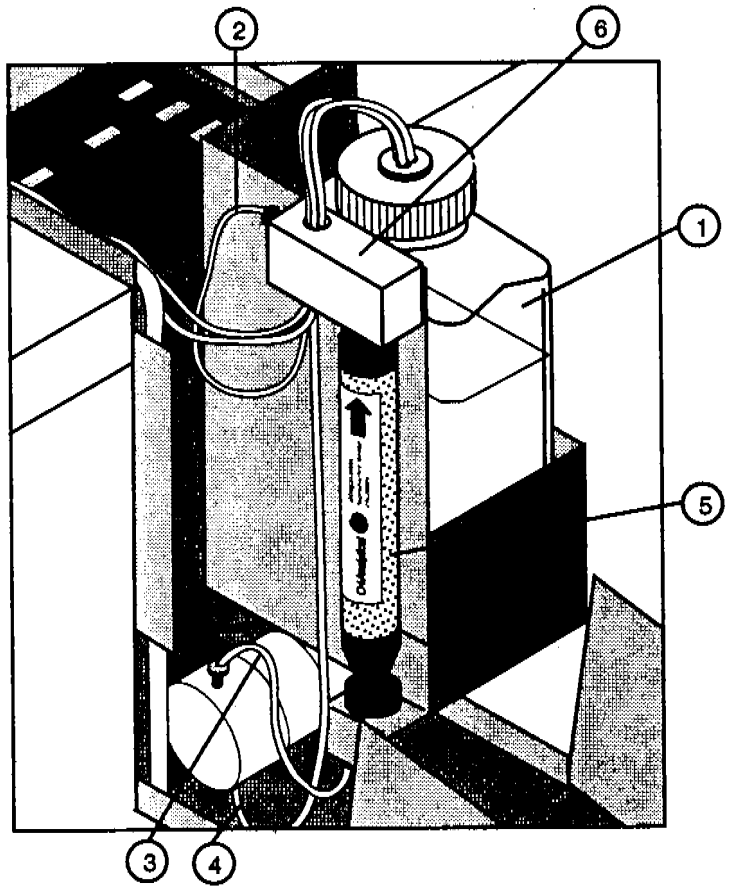
Transfer Line transfers reaction products from the Reactor to the Cell (utilizes Reverse Ferrule and Knurled Nut).

Solvent Return Line transfers waste solvent and gas to Solvent Reservoir connection (utilizes Reverse Ferrule and Knurled Nut).

Cell Inlet Line carries solvent from the solvent block to the Cell (utilizes Reverse Ferrule and Knurled Nut).

Assembly Ground (ground tab) is connected to the Cell Cover ground wire.

5220 Cell/Solvent Assembly - Side View



1. Solvent Reservoir
2. Solvent Bypass Line
3. Solvent Pump Inlet Line
4. Solvent Pump Outlet Line
5. Resin Cartridge
6. Solvent Flow Block

Figure 2.4. Cell/Solvent Assembly - Side View

Solvent Reservoir (500-mL bottle) contains the electrolyte solvent to be used. The electrolyte solvent must be appropriate to the mode of analysis.

Solvent Bypass Line carries excess solvent from the Solvent Block to the Solvent Reservoir. Only a small portion of the solvent pumped is required at the Cell.

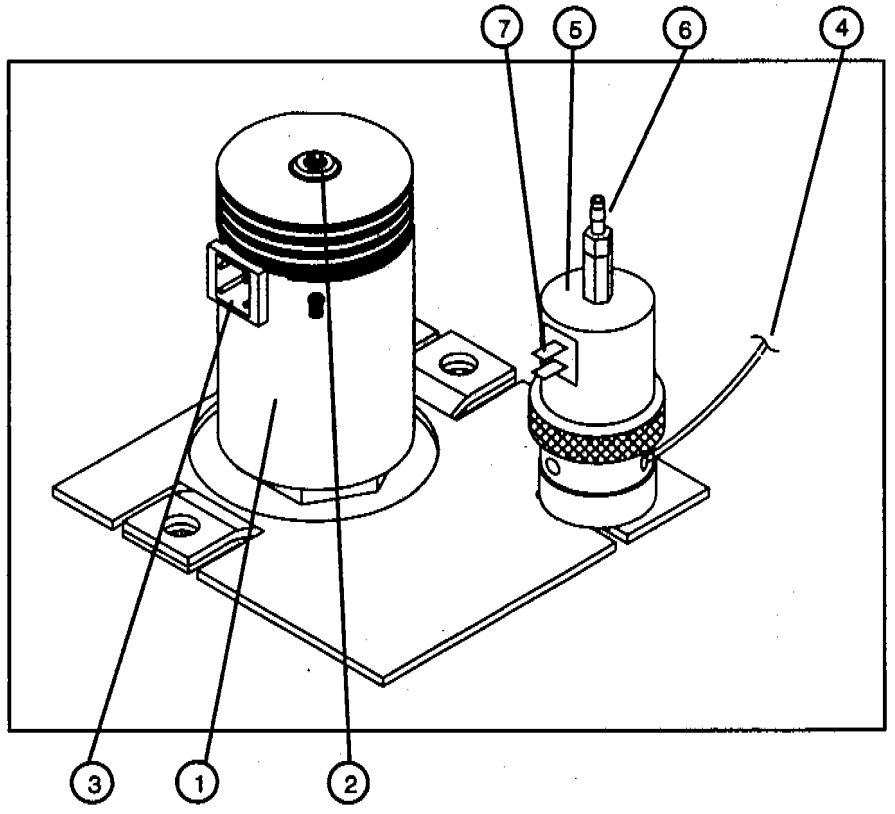
Solvent Pump Inlet Line carries solvent from the Solvent Reservoir to the Solvent Pump.

Solvent Pump Outlet Line carries solvent from the Solvent Pump to the Resin Cartridge.

Resin Cartridge contains two 10-micron filters and ion exchange resin. It filters the solvent, removing ions and particulate impurities.

Solvent Flow Block accepts solvent from the Resin Cartridge and splits it between the Solvent Transfer Line and Solvent Split Line. All connections are made with a Knurled Nut and a double-sided PEEK Ferrule.

5220 Reactor Assembly



1. Reactor
2. Reactor Union Assembly
3. Reactor Power Assembly
4. Reaction Gas Inlet Tube
5. Vent Valve
6. Vent Needle Valve
7. Vent Valve Power Connector

Figure 2.5. Reactor Assembly

Reactor heats the Reaction Tube to produce the conditions necessary for proper sensitivity to H, N, or S compounds. It contains a removable Reactor Core.

Reactor Base (not visible) provides a connection to the GC column, allows introduction of reaction gas (hydrogen or air), provides the Vent Line to the Vent Valve, and supports the Reactor.

Reaction Tube (not visible) is inserted into the Reactor and catalyzes the conversion of organic species to the corresponding ionizable gases. The tube used for the Halogen or Nitrogen Mode operation is nickel; for the Sulfur mode, an alumina tube is standard. Other materials may be used for specific applications.

Reactor Union Assembly seals the Reaction Tube to the Transfer Line that leads to the Cell by use of a GRP/VSP Ferrule on the Reactor end and a Teflon® (TFE) Ferrule on the Transfer Line end.

Reactor Power Connector (4-pin connector) mates with the Reactor Interface Cable and makes the power connection between the Cell Amplifier Board and the Reactor Heating Filament.

Reaction Gas Inlet Tube connects to a source of reaction gas (flowing at the rate specified for the mode of operation), and allows this gas to flow into the bottom of the Reactor through the Vent Valve Manifold.

Vent Valve, when shut (de-energized), passes reaction gas through both Vent Lines so that all gases entering the Detector Base flow through the Reaction Tube. When open (energized), the Vent Valve allows virtually all of the GC effluent to pass out the lower Vent Line instead of through the Reaction Tube.

Vent Line (not visible) connects to the Vent Valve Manifold so that unwanted GC effluents pass through the Valve instead of through the Reactor when the Vent Valve is open.

Vent Valve Manifold (not visible) introduces reaction gas to the Reactor Base, provides an exit pathway for the vent effluent, and supports the Vent Valve.

Vent Needle Valve (needle valve outlet fitting) can be adjusted to optimize the flow of electrolyte and gases through the Reactor and Cell Assembly when the Vent is open. It can also measure the total flow out to Vent. It should be set to 35-40 mL/min.

Vent Valve Power Connector joins with the Vent Cable. It makes the connection between the Vent Valve and Cell/Solvent System and, ultimately, to the power provided by the 5200 Detector Controller.

Chapter 3
Installation

In Chapter 2 the names and functions of the various components of the ELCD were defined. These names are used in this chapter to refer to components involved in the installation of the Detector onto a gas chromatograph.

Included with the 5220 is a Signal Cable for connecting the signal output of the Detector to a data output device. (Electrolyte is not supplied: electrolytes are specified in Table 4.2 of Chapter 4. All alcohol used for electrolyte should be ACS Reagent Grade quality.)

Stand-Alone 5220 ELCD or 5222 Dual ELCD Installation

Preparing GC

- Turn off GC power.
- Remove GC oven top, top right, right side, left side, and rear covers.
- Remove the selected Detector Port cover and insulation plug.
- Remove insulation inside Detector Port.

Installing the Reactor Base and Reactor

- Insert Reactor Base into appropriate Detector Port. (Do not remove top plastic plug from top of Reactor Base until ready to install Reaction Tube.)
- Align holes of Reactor Base with holes in top of GC and screw Detector Base to GC.
- Route Reaction Gas Line along top of GC using the existing tubing guides.
- Attach Gas Line from Reactor Base to port of Gas Flow Module on left side of GC.
- Install Reaction Tube and Brass Ferrule (Part #226456) into Detector Base by tightening Reactor Base Nut with the 1/4" Nut Driver (supplied in Start-up Kit). See Fig. 5.3. for the orientation of Brass Ferrule.
- Slide Reactor over Reaction Tube.
- Turn Reactor while slightly pressing downward, to insure proper seating of Reactor onto Reactor Base.
- Slide and properly seat Reactor Top Fitting (Part #227462), GRP/VSP Ferrule (Part #216358), and Reactor Union (Part #226472) over Reaction Tube and slightly tighten down. (see Fig. 5.1).

- Attach Reactor Power Assembly Connector (Part #231365) to Reactor as in Fig. 2.5.
- Attach Vent Valve Power Connector (Part #214890) to Reactor (confirm that the Vent is screwed down securely).
- Open GC oven door to install Column. Slide Column through Knurled Nut and GRP/VSP Tube Ferrule. (see Fig. 4.1).
- Remove bottom plastic plug from Detector Base.
- With one hand, push Column and GRP/VSP Ferrule up into Reactor Base until Column stops. With other hand, finger-tighten Knurled Nut in clockwise position. See Chapter 4, "Operation" for further details on installing Column, if necessary.
- Plug Detector Signal Cable (supplied with Detector) into Analog Input Board on top right side of GC.
- Plug in and route Remote Start Cable along the top to the back of the GC. Extend cable out of back so the end of the cable will be available after the cover is put back in place.
- Replace upper right and left side GC cover.

Installing the Cell/Solvent Assembly

- With the upper right GC cover put back in place, hang Cell/Solvent Assembly over right side of GC so that the tab on Assembly locks under the GC cover. (see Fig. 2.3).
- Attach Grounding Cable from Assembly to top of GC with supplied screw. (see Fig. 2.5).
- Remove top cover of Cell/Solvent Assembly by gently pulling upward.
- Plug in Reactor Power Connector (Part #231365) from the Reactor to the connector on the Cell Amplifier Board. (see Fig. 2.3).
- Plug in Vent Power Connector (Part #214890) from the Vent to the connector on the Cell Amplifier Board. (see Fig. 2.3).
- Plug in Ground Lead. (see Fig. 2.3).
- Plug in Cell Interface Connector (Part #216010). (see Fig. 2.3).
- Make a Transfer Line by cutting a minimum length of 1/16" x .20 I.D. TFE tubing that will extend from top of the Reactor to the Cell.
- Slide TFE Transfer Line through the 1/16" Male Knurled Nut (Part #226357) and a 1/16" TFE Ferrule (Part #156689). Note orientation of TFE Ferrule in Fig. 5.1.
- Finger-tighten 1/16" Male Knurled Nut and Transfer Line into the Cell as in Fig. 2.3.

- Connect other end of Transfer Line to the top of the Reactor by sliding tubing through the 1/16" Female Knurled Nut (Part #226357) and TFE Ferrule (Part #216358). Note direction of Ferrule in Fig. 5.1.

- Finger-tighten the 1/16" Female Knurled Nut and Transfer Line onto top of Reactor.

- Open the solvent enclosure by pulling outward on the side cover of the Cell/Solvent Assembly.

- Fill Solvent Reservoir with the appropriate solvent for the application (see Table 4.2).

- Replace the Solvent Reservoir lid.

Installing the Gas Flow Module

The Gas Flow Module is the gas control device that appears between the main gas source and the ELCD. The Gas Flow Module is mounted in the upper left corner of the HP 5890 Series II GC in the area marked "Detector A" or "Detector B."

The bottom, side port (Hydrogen) is the position used for the ELCD. The H_2 gas line from the Reactor Base is connected to this port after the Reactor Base is installed. (see Fig. 3.1).

- Slip the Gas Flow Module into place on the back side of the front left panel as shown in Fig. 3.1. The Module fits over the two stand-off posts and is fastened with the M4 x 45 mm Mounting Screw in the center location (**do not over-tighten the screw**). Check the ON/OFF valve for freedom of movement. If the valve knob is difficult to adjust, loosen the Mounting Screw and adjust the ON/OFF knob position until it is approximately half-open. Re-tighten the Mounting Screw and re-check the valve knob for freedom of movement.

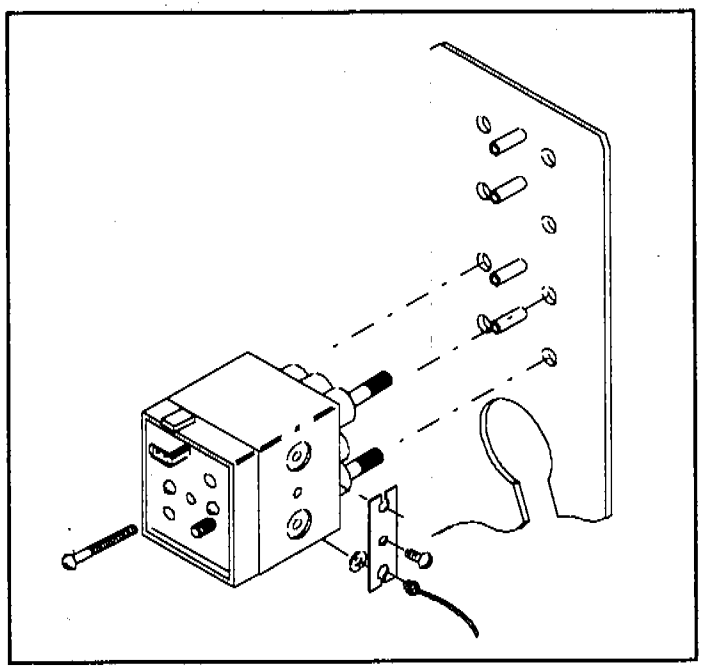


Figure 3.1. Gas Flow Module

- Remove the plastic protective backing to expose the adhesive on the ELCD Gas Flow Module face plate. Slip the plate over the valve knob on the front panel of the GC, and press it firmly into place.

- Carefully route the H_2 Gas Line from the installed Reactor Base toward the back of the GC, then along the left side (using the two clips provided), and toward the front of the GC until the Line reaches the slot provided.

- Attach gas supply lines to flow block by using the Manifold Plate (Part #197772) and Manifold O-Ring (Part #185116) as in Fig. 3.1.

Installing the 5200 Detector Controller

- Set 5200 Detector Controller in place (preferably to the right side of GC). (see Fig. 2.2 for rear back of Detector Controller.)

- Plug Power Cable into back of Detector Controller.

- Plug Power Cable into standard 110 VAC power outlet. **(Turn the power switvh off and set the voltage selector to the proper position)**

- Plug Cell Interface Cable from GC into Cell Interface Receptacle on back of 5200 Detector Controller and screw in both pins.

- Plug Signal Cable from GC into Signal Out Connector on back of 5200 Detector Controller.

- Plug in 15-Pin D-Sub Connector into I/O Port on back of Detector Controller.

NOTE: Remaining cable from 15-pin D-Sub Connector can be used to provide remote start to peripheral data device.

See Chapter 4, "Operation" for complete information on installing Columns, setting gas flows, filling Solvent Reservoir, and setting solvent flow.

Chapter 4
Operation

This chapter discusses the operation of the detector for analyzing samples. The 5220 can be operated in the Halogen, Sulfur, or Nitrogen Mode.

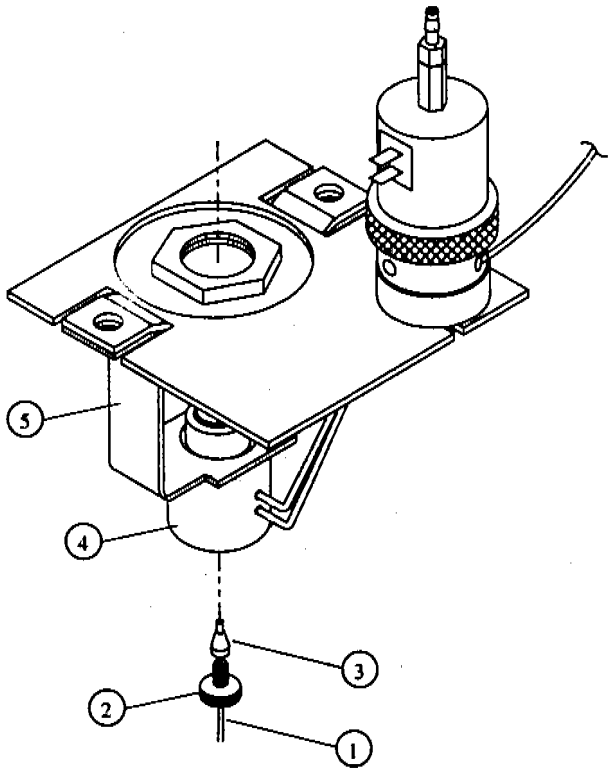
Install Column

WARNING: Flammable hydrogen gas and alcohol vapors will be present in the Solvent Reservoir during operation. Adequate steps should be taken for their ventilation.

The ELCD base is optimized for 0.53 mm and smaller I.D. capillary columns. To install the column into the detector base (see Fig. 4.1):

NOTE: These instructions are written for the 1/32" base. Installation of other bases is similar.

NOTE: If a positive flow of gas to the conductivity cell is not present (i.e., when the column is not installed), the solvent flow must be turned OFF. This will prevent solvent from backflushing into the Reactor and irreversibly fouling the reaction tube.



1. Capillary Column
2. 1/32" Knurled Column Nut
3. 1/32" GRP/VSP Tube Ferrule
4. 1/32" Reactor Base Assembly
5. Reactor Base

Figure 4.1. Column Installation

- Remove the 1/32" Nut from the Reactor Base Assembly.
- Slide the Column Nut onto the end of the Capillary Column.
- Slide a 1/32" GRP/VSP Ferrule onto the Column (with the tapered end facing toward the tip of the Column). See Table 4.1 for appropriate Ferrule.

Base	Column	Ferrule I.D.	Material I.D.
1/32	0.53	0.8 mm	GRP/VSP
1/32	0.32	0.5 mm	GRP/VSP
1/32	< 0.32	0.4 mm	GRP/VSP
1/16	0.53	0.8 mm	GRP/VSP
1/16	0.32	0.5 mm	GRP/VSP
1/16	< 0.32	0.4 mm	GRP/VSP
1/8	packed	—	GRP/VSP
1/4	packed	—	GRP/VSP

Table 4.1 Ferrule Specifications

- Clip a small section off the end of the Column to remove any foreign particles that may have lodged into the Column's open end. Use a proper Column cutting tool and check for a clean, straight cut.
- With the Ferrule and Nut on the Column, insert the Column end into the Base assembly while finger-tightening the Nut.
- Gently push the Column into the Base as far as it will go. Tighten the Nut approximately 1/4-turn past snug while exerting a slight upward pressure on the Column. Do not use a wrench or pliers on 1/32" Knurled Column Nut.
- Leak-check the connection.

Set Gas Flows

WARNING: Never adjust the Needle Valves to the point of complete shut-off to avoid inner-seal damage.

Gas flows are set by adjusting the Needle Valve at the center of the larger ON/OFF Valve located on the PID/ELCD Gas Flow Module. Use a small screwdriver to adjust the Needle Valves; the Valves open ***counterclockwise***.

NOTE: Do not use Nitrogen as a carrier gas.

WARNING: Flammable hydrogen gas and alcohol vapors will be present in the Solvent Reservoir during operation. Adequate steps should be taken for their ventilation.

To adjust the gas flows, follow these procedures:

- Ensure that the solvent flow is OFF (see [RATE] key operation in this chapter).
- Remove the Solvent Return Line from the Solvent Reservoir and attach a flowmeter to its end.
- Ensure the Vent Valve is closed (see [VALVE] key operation in this chapter).
- Set the Column Carrier Gas flow to the desired rate.

- Open the reaction gas ON/OFF Valve at the Gas Flow Module. Set reaction gas flow so that total carrier and reaction gas is 130±5 mL/min.

- For ELCD stand-alone operation, adjust the inner Needle Valve until the carrier-plus-hydrogen reaction gas flow equals 130mL/min.

- For Tandem PID/ELCD operation, open the Makeup ON/OFF Valve at the Gas Flow Module. Adjust the inner Needle Valve until the carrier-plus-makeup gas flow equals 130mL/min.

- For Tandem PID/ELCD operation, open the sweep gas ON/OFF valve and adjust the Lamp sweep gas flow until the column-plus-makeup-plus-sweep gas flow equals 130±5 ml/min.

NOTE: The PID Lamp sweep gas serves as the ELCD Hydrogen reaction gas.

- Remove the flowmeter from the Solvent Return Line and insert the line into the Solvent Reservoir as far as it will go.

- Leak-check connections, but ***do not*** use any liquid leak detectors around the Reactor Assembly and the Cell.

NOTE: The Vent Valve flow is set at a fixed rate, thus requiring no adjustment.

1. Vent Valve Manifold
2. Vent Valve
3. Outlet Restrictor

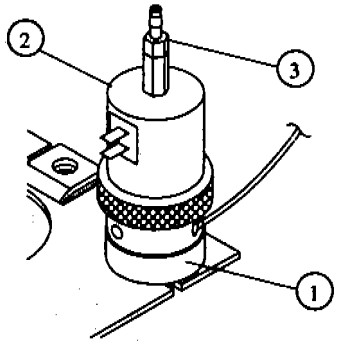


Fig. 4.2. Vent Valve

Fill Solvent Reservoir

The electrolyte in the Solvent Reservoir will slowly evaporate and must be refilled accordingly. Top-off the Reservoir with the following Solvent every 300 hours (approximately 14 days).

Mode	Solvent (Electrolyte)
Halogen	100% ACS Reagent Grade n-propanol (normal propyl alcohol)
Nitrogen	90:10 (v/v) 18 megohm-cm or better deionized, degassed water/ACS Reagent Grade t-butyl alcohol
Sulfur	100% ACS Reagent Grade methanol

Table 4.2. ELCD Electrolyte Solvent

NOTE: If the Solvent Reservoir empties completely, the solvent pump will run dry, accelerating wear of the internal gears in the pump head. Extended "dry" operation produces graphite gear particles at the pump outlet and causes an inability of the solvent pump to generate and maintain sufficient pressure for proper electrolyte flow.

Fill the electrolyte Solvent Reservoir with the appropriate solvent for the selected mode, as listed in Table 4.2. The Solvent Reservoir holds 500 ml of electrolyte. Refill the reservoir routinely (see Chapter 5) "Maintenance.".

Set Solvent Flow

Set solvent flow according to the following procedure:

- Remove the Solvent Reactor Line from the Solvent Reservoir and insert the end into a small vial or other container suitable for collecting solvent.

Mode	Rate	Percentage Setting
Halogen	25-40 µl/min	25-50%
Nitrogen	40-80 µl/min	35-65%
Sulfur	20-40 µl/min	10-40%

Table 4.3. Solvent Flow Rates

NOTE: The solvent rate versus percentage setting is not a linear relationship (i.e., a flow rate of 30 µL/min corresponding to a 25% flow setting does not necessarily mean a flow rate of 60 µL/min equals a 50% flow setting). The same percentage setting may also yield varying flow rates for different modes because of different I.D. tubing.

- Turn the solvent flow to ON and set the flow percentage according to Table 4.3.
- Collect solvent in the small vial for 3—5 min.
- Measure the solvent by using a microliter syringe and determine the microliter per minute rate.

- Adjust the percentage setting until a flow rate is within the range given in Table 4.3.
- Return the Solvent Return Line to the Solvent Reservoir. Place the end of the line above the solvent level to reduce solvent evaporation and back pressure on the Cell.
- Check for solvent leaks.

The solvent flow rates given in Table 4.3 ensure reliable ELCD response. Generally, lower solvent flow rates increase Detector response but also increase baseline noise. Using Table 4.3 as a guide, adjust the solvent flow rate to maximize signal-to-noise ratio for any specific application.

Once a desired solvent flow has been set, that rate generally can be reachieved by resetting the solvent percentage after various testing or maintenance procedures. The Detector response is not dependent on exact repeatability of electrolyte flow rate. Periodically, check the actual solvent flow rate by collecting solvent from the Solvent Return Line.

Set GC Settings

Set the GC temperature program settings to the desired values. Do not set a temperature for the Detector Base if operating a stand-alone ELCD. The 5520 ELCD base "tracks" or follows at approximately 30°C above the GC oven temperature. This feature minimizes column bleed.

Set 5220 Setpoints

The 5220 setpoints are controlled through the **5200 Detector Controller**. Use Table 4.4 as a guide to 5220 operation parameters (see the **"5200 Detector Controller Operation"** section of this chapter for proper 5200 keypad sequences).

Control	Active Key	State Value
Mode Selection	[DET]	Turn ON proper sensitivity
Output	[SIG]	ON
Zero Offset	[ZERO]	Determined by background noise
Reactor	[TEMP]	ON
Rx Temperature	[TEMP]	
Halogen Mode		850-950°C Volatile Organics
		900-1100°C Pesticides
		900-1100°C PCBs
		800-1100°C Semi-Volatiles
Sulfur Mode		800-1100°C
Nitrogen Mode		800-1100°C
Vent Valve	[VALVE]	ON (open) 0.01 min (typical)
		OFF (close) end of solvent peak
Solvent Flow	[RATE]	ON
Solvent %	[RATE]	See Table 4.3

Table 4.4. 5220 Operation Parameters

Operational Guidelines

- Do not use N_2 as carrier or makeup gas.
- Perform periodic maintenance (see Chapter 5) "Maintenance.".
- Always use the Vent Valve to vent the solvent. Solvent injected without venting may immediately and irreversibly foul the Reaction Tube, causing severe peak tailing and loss of response.
- Do not use solvents that contain halogens, sulfur, or nitrogen. If possible, also avoid solvents that contain oxygen.
- The ELCD has been designed to be left ON during standby periods. Repeatedly turning the power OFF and ON to conserve gas will increase warm-up time, and possibly foul the Reaction Tube or cause Reactor failure.
- Once a desired electrolyte flow has been set, that rate generally can be reachieved by resetting the solvent percentage after various testing or maintenance procedures. The detection response is not dependent on exact repeatability of electrolyte flow rate.
- Use the highest purity gases available (99.999%) for best signal-to-noise ratio. Proper gas purity and conditioning is crucial for successful ELCD operation.
- Replace Reaction Tube, resin, and Solvent Lines when switching operating modes.

5200 Detector Controller Operation

The 5200 Detector Controller can control up to two detectors — two ELCDs, two PIDs, or one ELCD and one PID. Both the PID and the ELCD operation are monitored and controlled through the 5200 keypad display.

The **5200 Detector Controller** keypad display consists of an alphanumeric and instrument status display.

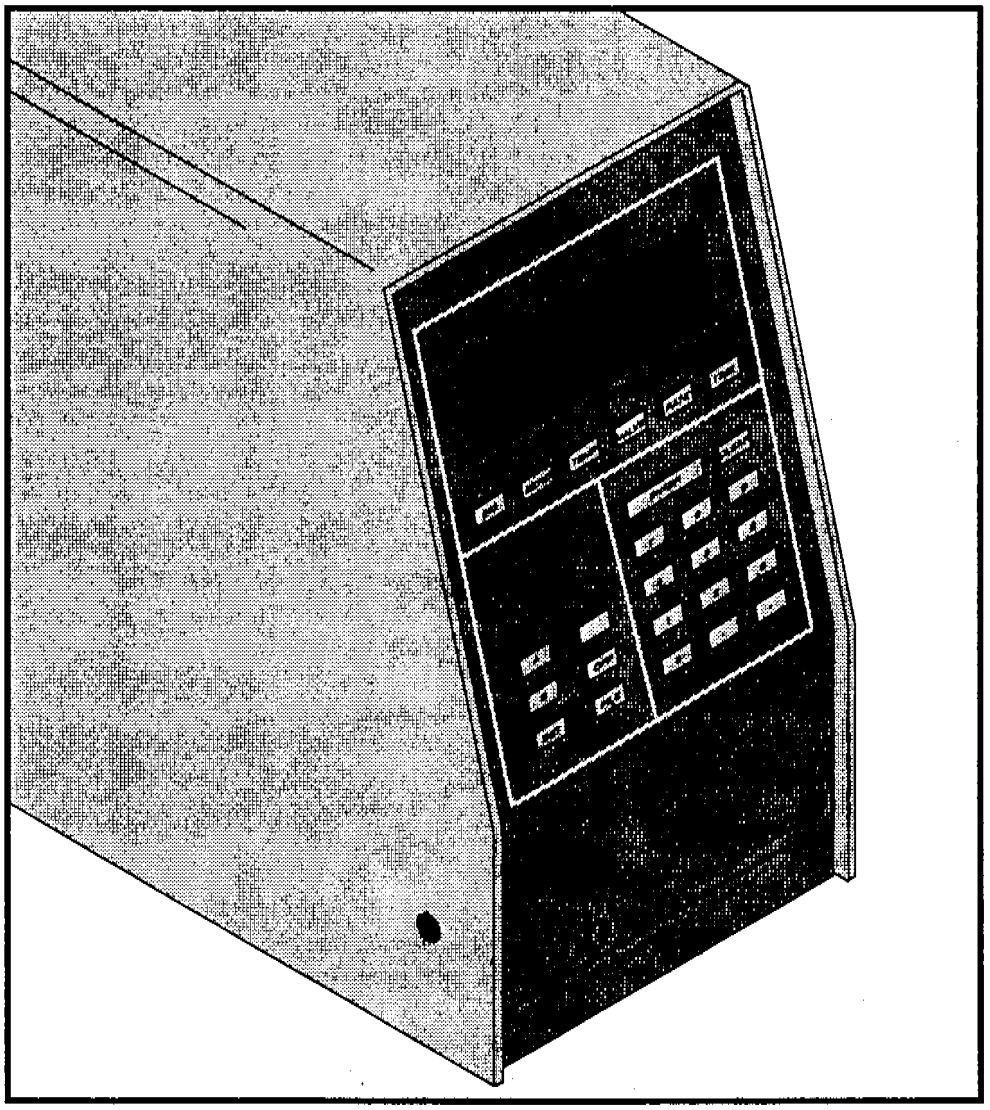


Fig. 4.3. 5200 Detector Controller Keypad

Alphanumeric Display shows current Detector setpoint values, actual Detector values that are continuously monitored, and general Detector information/diagnostic messages.

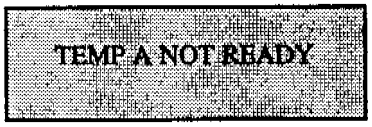
Detector Status Display shows overall Detector ready status and indicates when a Detector is in a run mode.

Not Ready Display (red) indicates the status of the Detector(s) functions controlled by the 5200.

The Not Ready Display will light up if:

- Detector itself is turned OFF;
- Detector signal is turned OFF;
- ELCD Reactor temperature is turned OFF or is adjusting to a new value;
- ELCD Solvent Flow is turned OFF or is adjusting to a new value; or
- PID Lamp Current is turned OFF or is adjusting to a new value.

To identify one or more detector functions causing a NOT READY display, press the [CLEAR] key repeatedly, scrolling through various screens. For example, if Reactor A temperature is OFF or adjusting to a new setpoint, the NOT READY display would be illuminated. Pressing the [CLEAR] key would yield the following display.



Temp Not Ready Display

If the NOT READY display is ***not*** illuminated, the 5200 is ready.

Active Keys

The 5200 Detector Controller has eight keys that are active from the "OI System Ready" display. The eight active keys are [SIG], [ZERO], [TEMP], [VALVE], [RATE], [TIME], [DET], and [FILE]. To display, alter, or input values on the keypad, one of these eight keys is pressed at the beginning of a key sequence.

Modifier Keys

Each Detector value may need to be modified using the [A], [B], [ON], or [OFF] keys after pressing the appropriate Active key.

The 5200 assigns each Detector to a specific channel, channel A or B. After pressing one of the Active keys, the system will display channel A values. Detector B can be accessed by pressing the [B] key. If a Detector is not installed in a channel, the display will exhibit a "NOT INSTALLED" message. For example, pressing [SIG] [B] would give the display "DETECTOR B NOT INSTALLED" if no Detector was installed in channel B. If Detectors are installed in both channels, the [A] and [B] keys can be used to alternate between display information for each Detector.

[ON]/[OFF] Keys

The [ON] and [OFF] keys are used to manually turn the Detector and Detector functions on and off.

Input Keys

After pressing an Active key, enter setpoints by using the 10 number keys, the decimal [.] key, and the minus [-] key.

For a numeric value, the display shows a flashing asterisk (*) while entering a new setpoint. The asterisk disappears upon pressing the [ENTER] or [CLEAR] key.

After entering a number, press the [ENTER] key to accept the setpoint. When [ENTER] is pressed, the setpoint value is verified and, if valid, becomes the new setpoint for the function. If the setpoint entered is invalid (i.e., out of range), an appropriate message is displayed. A valid setpoint can then be entered. The original setpoint is retained until a new valid setting is entered.

To erase an entry, press the [CLEAR] key after entering the number but before pressing the [ENTER] key. The asterisk disappears and the original setpoint is retained. Pressing the [CLEAR] key at any other time changes the 5200 screen display to "OI SYSTEM READY" or shows the reason the system is not ready (see "Detector Status Display" earlier in this chapter).

Key Operation

[SIG] Key

Display Signal Output

Pressing the [SIG] key followed by the appropriate channel key will display the Detector signal output and the functions ON/OFF state for each 5200 channel assigned to an ELCD. The signal output ranges from ±999.999 mV.

Signal Output Display

NOTE: If a single PID is installed to a channel in the 5200, the signal output is controlled and monitored through the GC. If the PID is installed in Channel B, the 5200 will ignore a [SIG], [B] request.

Turn Signal ON/OFF

[SIG], [ON], or [OFF]

If the Detector signal is turned OFF, the signal output "OUT" will display a zero mV reading. Turning the Detector signal OFF does not turn off the Detector itself. The screen will display "SIG A (or B) OFF" when the [CLEAR] key is pressed, if the signal is OFF.

[ZERO] Key

Display Zero Offset

Pressing the [ZERO] key displays the current zero offset value and the resulting signal output. The signal output will not be displayed if the Detector is turned OFF. The zero

offset value subtracts a constant value from the Detector signal. The zero function can increase the dynamic range by subtracting the background signal from sources such as impurities in the supply gasses and column bleed.

Adjust Zero Offset

[ZERO], [1], [0], [.], [5], [ENTER]

The above setting will adjust the Detector signal output by *subtracting* a constant 10.5 mV from the output. To add to the signal or shift the baseline upward, a negative value is entered. A value between ±999.999 can be entered. To return to the original signal output, set the zero offset value to zero. All zeroing adjustments should be performed during quiet baseline activity, (i.e., not during a run). All attempts to correct any background noise should be performed before using this function.

ZERO A	10.5mV
Out A	-10.500mV

Zero Offset Display

Turn Zero Offset ON/OFF

[ZERO], [ON], or [OFF]

Adjust Zero Offset with Autozero

[ZERO], [SIG], [ENTER]

The autozero function adjusts the current detector signal output to zero by subtracting a constant value from the output.

[TEMP] Key

Display Reactor (Rx) Temperature

Pressing the [TEMP] key displays the Reactor temperature setpoint and the actual Reactor temperature for each ELCD installed in the 5200.

Turn Reactor Temperature ON/OFF

[TEMP], [ON], or [OFF]

Adjust Reactor Temperature

[TEMP], [9], [0], [0], [ENTER]

The above sequence will change the Reactor temperature to 900° C. Temperatures from 400 to 1100° C are valid. The screen will display "TEMP A (or B) NOT READY" under the [CLEAR] key if the Reactor is OFF or is adjusting to a new temperature.

TEMP Rx A	900° C
Actual	450 °C

Reactor Temperature Display

[VALVE] Key

Display Valve State

Pressing the [VALVE] key displays the state ON (open) or OFF (closed) of the Vent Valve. The Vent is typically used to eliminate solvents entering the ELCD Reactor. Alkane solvents (e.g., hexane) produce unreacted hydrocarbons and elemental carbon that interfere with Detector response. Oxygenated solvents (e.g., methanol) irreversibly foul the Reaction Tube.

Manual Vent Operation

Open Vent: [VALVE], [ON]
Close Vent: [VALVE], [OFF]

Open Vent Display

Set Vent Program

Vent Open Time: [VALVE], [ON], [.], [5], [ENTER]
Vent Close Time: [VALVE], [OFF], [3], [ENTER]

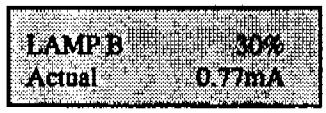
The above key sequences open the Vent at 0.50 minutes and close the Vent at 3.00 minutes. Values from 0 to 999.99 minutes can be entered. The typical setting for venting the solvent is to set the open time to zero and set the close time to correspond with the end of the solvent peak (usually approximately 3 minutes).

NOTE: The vent is normally open while deactivated.

[RATE] Key

Pressing the [RATE] key will either display the ELCD solvent flow rate or the PID Lamp intensity setting, depending on the specific Detector assignment to each channel.

ELCD Solvent Flow Rate



PID Lamp Intensity Setting

Display Solvent Flow

Pressing the [RATE] key displays the solvent flow as a percentage for the assigned ELCD channel(s).

Turn Solvent Flow ON/OFF

Channel: [RATE], [ON], or [OFF]

The screen will display "FLOW A (or B) NOT READY" under the [CLEAR] key if the flow is turned OFF.

Adjust Solvent Flow

[RATE], [5], [0], [ENTER]

The above key sequence adjusts the solvent flow to 50%. A value from 0 to 100% can be entered, whether or not the solvent flow is ON or OFF. To measure the actual µl solvent flow, collect solvent in a small vial exiting the Solvent Return Line during a timed period. The timer built into the 5200 is useful for this purpose (see "Timer Operation" in this chapter). Measure the solvent by drawing it into a µl syringe. Typical flow rates and their corresponding percentage settings for each mode are listed in Table 4.2.

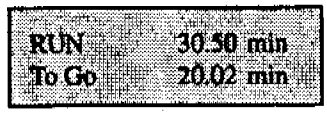
[TIME] Key

The internal timer in the 5200 is controlled through the [TIME] key. Specific run times can be preset for timed events.

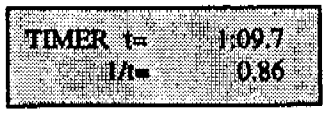
Display Time Functions

Repeatedly pressing the [TIME] key displays the remaining "to go" run time and a stopwatch timer function. Other instrument functions can be accessed through the appropriate Active key without stopping or resetting the timer. The timer continues to run, but it is not displayed until the [TIME] key is pressed again.

Elapsed Time Display



"To Go" Time Display



Timer Display

Set Run Time

[TIME], [3], [0], [.], [5], [ENTER]

The above entry sets the 5200 run operation time to 30.50 minutes. Run times up to 999.99 minutes are valid.

If an external remote start is ***not*** connected to the 5200, the 5200 run time should be set to match that of the GC.

START/STOP Run Timer

Start: [TIME], [ON] or [ENTER]
Stop: [TIME], [OFF]

Pressing the [ON] (or [OFF]) key starts (or stops) the run timer and timed events. Stopping the run does not reset the timer.

Clear (Reset) Run Timer

[TIME], [CLEAR]

NOTE: If the Run Timer is active (ON), pressing the [CLEAR] key only resets the timer to zero, it does not turn the timer OFF.

The 5200 will automatically start for each manual or remote start from the GC. At each external start, the 5200 Run Timer automatically starts at zero.

NOTE: If the GC run is stopped, the internal timer in the 5200 continues. The 5200 can also be stopped by using the [DET] and [OFF] keys.

If a sequence table from a data-handling system is used utilizing various run times:

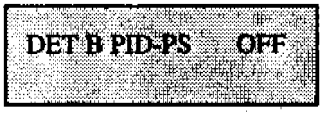
- an external remote start to the 5200 must be used; and
- the run time on the 5200 must be set to a higher value (e.g., 999 minutes) than the longest GC run.

The [TIME] key also has a stopwatch timer mode. Both time (to 0.1 second) and inverse time (to 0.01 min^{-1}) are simultaneously displayed. Use the [ENTER] key to start and stop the stopwatch. Use the [CLEAR] key to reset the stopwatch to zero. The stopwatch timer function is independent of the RUN timer and continues running while other Active keys are pressed.

[DET] Key

Pressing the [DET] key displays the ON/OFF state for either the PID or the ELCD. It also displays the high or low sensitivity selection and the Sulfur, Nitrogen, or Halogen Mode for the ELCD.

Detector Display for ELCD



Detector Display for PID

To turn Detector ON/OFF, press [DET], [ON], or [OFF]

When the Detector is turned OFF:

- the ELCD signal output, reactor, and pump are turned OFF;
- the PID high voltage (lamp and polarizing) are turned OFF; and
- the "NOT READY" display lights up and the message "DET A (or B) DISCONNECT" is displayed when the [CLEAR] key is pressed.

[FILE] Key

The [FILE] key is used to GET, SAVE, and DELETE (DEL) Detector setpoints controlled by the 5200. Upon pressing [FILE], the file menu is displayed. GET, SAVE, and DEL can be accessed by selecting the appropriate input number key.

File Menu Display

The 5200 has 14 file numbers for Detectors A and B that can store files containing Detector setpoints. Files are either undefined (no name and no setpoints are stored under a file number) or defined (a name and setpoints are stored under a file number).

SAVE FILE

Setpoints can be saved under a specific name by pressing [FILE] [2]. The "SAVE TO FILE" display appears, and a file can then be selected by scrolling with the [ON] or [OFF] keys. Saving new setpoints over an existing file replaces all setpoints currently in the selected file.

Once the file is selected, it is named and then saved by one of the following three methods:

- Use the 12 input keys ([0] to [9], [.], and [-]) to name an undefined file or to rename an existing file, then press [ENTER] to save the setpoints. If a file is named but not saved within 5 seconds, the 5200 defaults to the file menu display.
- Use the [ENTER] key only to name and save a file as its file number if the file is undefined. For example, selecting the undefined file number 7 followed by [ENTER] would save the file as "FILE 7."
- Use the [ENTER] key only to retain a file name for a previously named file. For example, selecting the existing file "2: 601/602" followed by [ENTER] retains the file name and replaces all current setpoints under file "2: 601/602."

When using the [ENTER] key to save a file, one of two messages will be displayed. The message "FILE SAVED" indicates that the new setpoints were saved under the selected file. The message "FILE NOT CHANGED" indicates no setpoints were changed to the selected file.

GET FILE

Saved files can be retrieved by pressing the [FILE] key followed by the [1] key. Select a file scrolling with the [ON] and [OFF] keys. Press [ENTER] to load a selected file, or press [CLEAR] to return to the file menu. One of the following three messages will be displayed upon pressing [ENTER]:

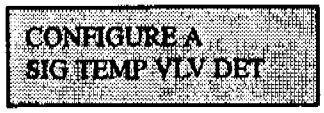
- "FILE LOADED" - The selected file setpoints have been successfully loaded and are now current.
- "NO FILE HERE" - A file was selected that contained no setpoints (i.e. an undefined file).
- "SAVE CURRENT SETTINGS? Y/N" - Indicates setpoints have been changed but not saved. Use the [ON] and [OFF] keys to select either Y (yes) or N (no) followed by [ENTER]. Selecting N ignores the changed setpoints and loads the selected file. Selecting Y allows the changed setpoints to be saved under a new or existing file. The screen display changes to the SAVE FILE mode (see *SAVE FILE*, above). Once the setpoints are saved, the selected file is loaded.

DEL FILE

File setpoints can be deleted using the [FILE] key followed by the [3] key. Select a file using the [ON] and [OFF] keys, then press [ENTER]. The file will then become undefined (i.e., the file name and all setpoints are erased).

Configuration Menu

Parameters can be changed for the SIG, TEMP, VALVE, and DET options by first pressing [CLEAR] [.]. The following menu will display:



Scroll through the options by pressing the [ON] or [OFF] key; select the desired function by pressing [ENTER] when that option is flashing.

SIG Option

The Linearization function is an internally programmed polynomial regression that allows the operator to predict linearity for the nitrogen or sulfur modes, which normally have a non-linear relationship. Please use suggested parameter settings to ensure linearity. The Smoothing function is used to eliminate any high frequency noise generated by normal ELCD operation, without affecting the signal output.

Select the SIG option to change the Linearization and/or Smoothing parameters. The following screen will appear:

Toggle between Linearization and Smoothing by pressing [ENTER]. Select ON or OFF by pressing the [ON] or [OFF] key.

TEMP Option

Select the TEMP option to turn the Reactor Calibration ON or OFF. The following screen will appear:

Select ON or OFF by pressing the [ON] or [OFF] key.

VLV Option

Select the VLV option to set the desired valve logic. The following screen will appear:

Select ON or OFF by pressing the [ON] or [OFF] key.

DET Option

Select the DET option to change the Detector's Sensitivity and/or Mode parameter settings. The following screen will appear:

Toggle between Sensitivity and Mode by pressing [ENTER]. Select the desired parameter by pressing the [ON] or [OFF] key. Sensitivity can be set to HI for Pesticides Analysis or LO for Purge-and-Trap Analysis. Mode can be set to H for Halogen, N for Nitrogen, or S for Sulfur.

Keyboard Lock

The keyboard can be locked to prevent settings from being changed. To lock the keyboard, press [CLEAR] [.] [-] [ENTER]. The following screen appears:

Press [ON] to turn the lock ON. Once the lock is on, any attempts to change Detector settings cause the message "Keyboard locked" to appear. Unlock the keyboard by pressing [CLEAR] [.] [-] [ENTER] and [OFF].

Chapter 5
Maintenance

Chapter 5 describes the scheduled and non-routine maintenance of the detector.

Scheduled 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 (see Table 5.1). Scheduled hours refer to number of hours of operation.

An instrument log book to record instrument operation time and document periodic maintenance is recommended. This log book can be used to record results of inspections and component replacement necessary for proper maintenance of the instrument.

WARNING: All servicing must be performed by qualified service personnel.

Maintenance Item	Schedule
Solvent Reservoir Refilling	300 hours
Resin Cartridge Replacement	700 hours
Transfer Line Rinsing	daily

Table 5.1. Routine Maintenance Schedule

Refilling Solvent

NOTE: If the Solvent Reservoir empties completely, the Solvent Pump will run dry, accelerating wear of the internal gears in the Pump Head. Extended "dry" operation produces graphite gear particles at the Pump Outlet and causes an inability of the Solvent Pump to generate and maintain sufficient pressure for proper electrolyte flow.

The electrolyte in the Solvent Reservoir will slowly evaporate and must be refilled accordingly. Top-off the Reservoir with the following solvent every 300 hours (approximately 14 days).

Mode	Solvent (Electrolyte)
Halogen	100% ACS Reagent Grade n-propanol (normal propyl alcohol)
Nitrogen	90:10 (v/v) 18 megohm-cm or better deionized, degassed water/ACS Reagent Grade t-butyl alcohol
Sulfur	100% ACS Reagent Grade methanol

Table 5.2. ELCD Electrolyte Solvent

Replacing Resin Cartridge

The Resin Cartridge performs the ion removal from the electrolyte and traps any particles produced from the Solvent Pump. Replace every 700 hours (approximately 1 month) according to the following procedure:

- Turn the solvent flow OFF.
- Remove the Resin Cartridge from the holder by pushing down on the Resin Cartridge to depress the Resin Plunger.
- Insert a new Resin Cartridge with the arrow pointing upward. Note the expiration date on the Resin Cartridge.
- Remove the Solvent Return and Bypass Lines from the Solvent Reservoir. If necessary, see Fig. 2.4.
- Set the solvent flow to 100% to increase the Pump speed.
- Turn the solvent flow ON and allow 50-100 mL of solvent to flow through the system while draining both the Solvent Return and Bypass Lines to waste.
- Replace the Solvent Return and Bypass Lines to the Solvent Reservoir and refill the Reservoir with the appropriate solvent. Refer to *Filling Solvent Reservoir* in this chapter, for proper solvent selection.
- Reset the solvent flow to the desired level.
- Check for solvent leaks around the ends of the Resin Cartridge. If necessary, adjust the Resin Cartridge.

Transfer Line Rinsing

The Transfer Line can become contaminated with decomposition products that exit the Reactor (usually unreacted hydrocarbons). The result can be peak tailing, baseline noise, and/or reduced response. Every 700 hours (approximately 1 month) or as these symptoms appear, rinse the Transfer Line as described in the following procedure.

NOTE: The Reactor should remain ***hot*** while this procedure is performed.

- For Halogen Mode, unscrew the Knurled Reactor Nut at the top of the Reactor (see Fig. 5.1).
- For Nitrogen/Sulfur Modes, disconnect the PEEK tubing at the Scrubber Union by loosening the Scrubber Tube Nut (see Fig. 5.2).
- With the solvent flow ON, allow the Transfer Line to sag to an open area on the top of the GC and away from the Reactor. Let solvent from the Solvent Reservoir backflush the Line onto a paper towel (it takes a few seconds for the solvent to reach the end of the Line). The solvent flow can be increased to facilitate this process.
- After several drops (approximately 10-20 µL) flow from the Transfer Line, turn the solvent flow OFF and raise the end of the line above the Cell, allowing the excess solvent to flow back into the Cell.

1. Reactor Casing
2. 1/16" Allen Screw
3. Reactor Core #214163
4. 4-Pin Meldin Reactor Connector
5. Reactor Top
6. 1/16" Reactor Top Fitting #227462
7. 1/16" GRP Tube Ferrule #216358
8. Reactor Union #226472
9. 1/16" TFE Ferrule #156689
10. 1/16" Knurled Reactor Nut #226357
11. 1/16" O.D. x 0.020 I.D. TFE Transfer Line #165896 (Halogen Mode)

NOTE: The above part numbers may be for single items only. Refer to OI Analytical for appropriate multiple packs (i.e. 5 pk, 6 pk, 10 pk).

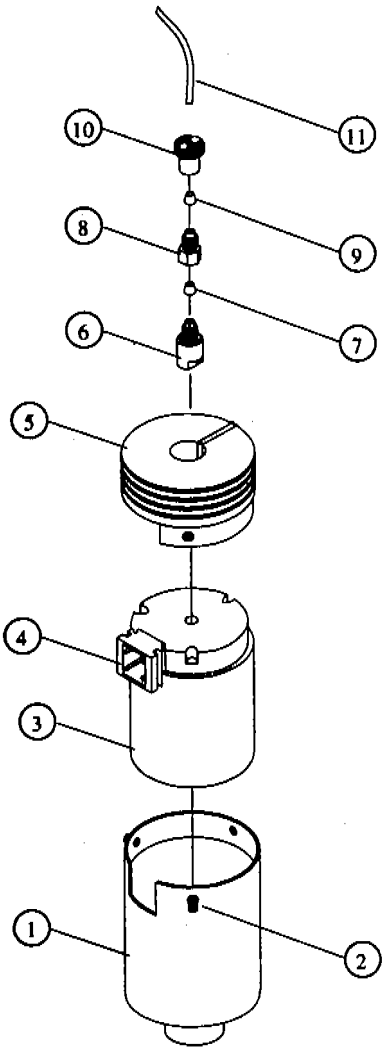
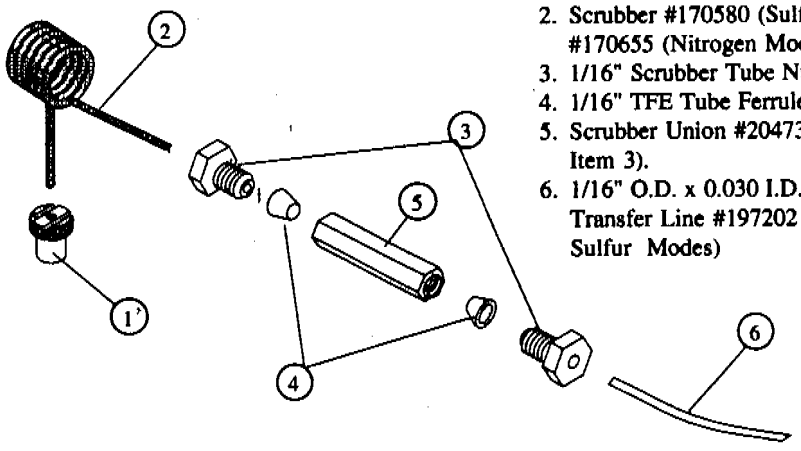


Figure 5.1. Reactor Assembly



1. 1/16" Knurled Reactor Nut #226357
2. Scrubber #170580 (Sulfur Mode); #170655 (Nitrogen Mode)
3. 1/16" Scrubber Tube Nut
4. 1/16" TFE Tube Ferrule #177626
5. Scrubber Union #204735 (Includes Item 3).
6. 1/16" O.D. x 0.030 I.D. PEEK Transfer Line #197202 (Nitrogen/ Sulfur Modes)

Figure 5.2. Scrubber Assembly

- Examine the end of the Transfer Line for restrictions or deformations caused by an overtightened Nut. If necessary, trim the end of the Line ensuring a clean, straight cut. Examine the TFE Ferrule used to seal the Transfer Line. Replace if necessary. Use a TFE Ferrule (Part #156689) for Halogen mode or a TFE Tube Ferrule (Part #177626) for Nitrogen and Sulfur Modes.

- Reconnect the Transfer Line to the top of the Reactor (Halogen Mode) or the Scrubber Union (Nitrogen/Sulfur Mode) and turn the solvent flow ON. Do not overtighten the nut — ***finger-tight*** will suffice.

If no improvement is observed, the Transfer Line should be replaced. Replace the Halogen Mode Transfer Line with approximately 10 inches or less of 1/16" x 0.020" I.D. TFE tubing (Part #165896). Replace the Nitrogen or Sulfur Mode Transfer Line with approximately 4 inches or less of 1/16" x 0.030" I.D. PEEK tubing (Part #197202).

Non-Scheduled Maintenance

Replacing Reaction Tube

CAUTION: The reaction tube, reactor, and supporting fittings may be hot.

To replace a Reaction Tube, follow the steps listed below (see Figs. 5.1, 5.2, and 5.3 when necessary):

- Turn the Reactor temperature and solvent flow to OFF.

- Allow the Reactor to cool to the point where it can be safely touched.

- Disconnect the Transfer Line by removing the Knurled Reactor Nut (Halogen Mode) or the Scrubber Tube Nut at the Scrubber Union (Nitrogen/Sulfur Modes).

NOTE: A Transfer Line backflush can also be performed at this time. Refer to *Transfer Line Rinsing,* in this chapter.

- Remove the Scrubber by removing the Reactor Nut (Nitrogen/Sulfur Modes).

- Remove the Reactor Union (Part #226472), GRP/VSP Tube Ferrule, and Reactor Top Fitting from top of the Reactor.

- Slide the Reactor off the Reaction Tube **(CAUTION: Reactor may still be hot).**

- Remove the Reaction Tube using 1/4" socket wrench (supplied in Start-up Kit) by sliding hollow wrench over the Reaction Tube and Reactor Base Nut and turning counterclockwise.

- Slide a new 1/16" Brass Ferrule (nose down), then either the Base Nut (stand-alone ELCD) or the Adapter Nut (Tandem PID/ELCD) over a new Reaction Tube. Use a Nickel Reaction Tube (Part #170522) and a 1/16" Brass Ferrule (Part #223776) for the Halogen/Nitrogen Modes or an Alumina Reaction Tube (Part #204719) and a 1/16" Brass Ferrule (Part #223776) for the Sulfur Mode.

- Install the Reaction Tube (with Nut and Ferrule) into the Reactor Base or PID Manifold (Tandem PID/ELCD) and firmly tighten the Nut to ensure a tight seal.

1. Capillary Column
2. 1/32" Column Unit
3. 1/32" GRP/VSP Tube Ferrule
4. 1/32" Reactor Base Fitting Assembly
5. GRP/Meldin Reactor Base Seal #223933
6. Reactor Base #216820
7. Reactor Base Lock Nut #214817
8. 1/16" Brass Tube Ferrule #226456 (Halogen/Nitrogen Modes)
   1/16" GRP/VSP Tube Ferrule #175910 (Sulfur Mode)
9. 1/16" Reactor Base Nut
10. Nickel Reaction Tube #170522 (Halogen/Nitrogen Modes)
    Alumina Reaction Tube #204719 (Sulfur Mode)
11. Vent Valve Manifold
12. Hydrogen Gas Line IN
13. 3-way 12 VAC Vent Valve #179325
14. Vent Valve Restrictor
15. Reactor

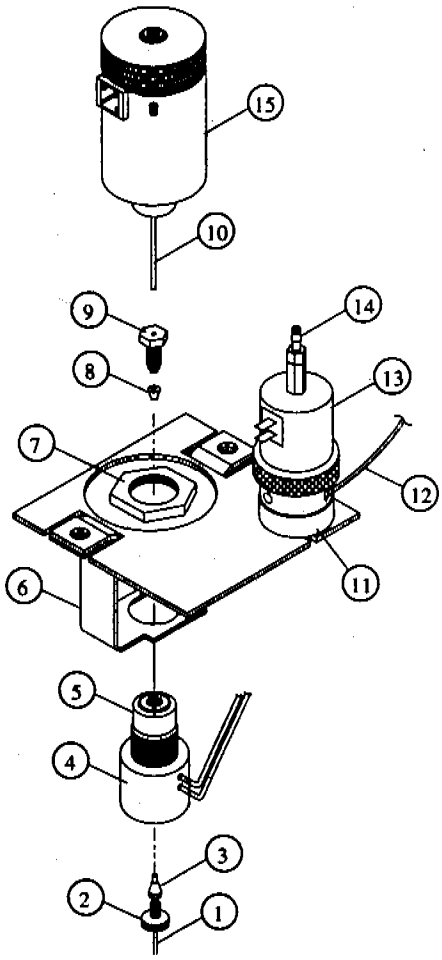


Figure 5.3. Reactor Base Assembly

- Install the Reactor by sliding it over the Reaction Tube. Seat the Reactor into the Nut by a slight downward side-to-side motion. The Reactor can be seated in one of six positions to best facilitate routing the Cable that is attached to the 4-pin Connector.

- Replace the 1/16" GRP/VSP Tube Ferrule (Part #206524) at the top on the Reactor.

- Turn the Reactor ON and set the temperature to 950°C for approximately 10 minutes to "condition" the Reaction Tube.

- After conditioning the Reaction Tube, properly seat Top Fitting onto top of Reactor and replace the Reactor Union. Tighten the Reactor Union securely against the Reactor Top Fitting.

- For Halogen Mode, attach the Transfer Line by ***finger-tightening*** the Knurled Reactor Nut onto the Reactor Union. Examine the end of the Transfer Line for restrictions or deformations caused by an over-tightened Reactor Nut. If necessary, trim the end of the Transfer Line to ensure a clean, straight cut. Examine the 1/16" TFE Ferrule (Part #156689) seated in the Reactor Nut. Replace if necessary.

- For Nitrogen/Sulfur Modes, attach the Scrubber by inserting the correct end into the Reactor Nut with the 1/16" TFE Ferrule (Part #156689). See Fig. 5.2 for the proper Scrubber orientation. The Reactor Nut needs to be only ***finger-tight.*** Examine the TFE Ferrule and replace, if necessary.

- For Nitrogen/Sulfur Modes, attach the PEEK Transfer Line onto the Scrubber Union using the 1/16" TFE Tube Ferrule (Part #177626). The Scrubber Tube Nut should only be ***finger-tight.*** Inspect the Ferrule and replace if necessary.

- Check for leaks, but do not use any liquid leak detectors around the Reactor.

- Turn the solvent flow to ON, adjust the Reactor temperature to the desired level, and allow the system to stabilize (approximately 5-10 minutes).

CAUTION
The reactor is hot!

The 1/16" TFE Ferrule (Part #156689), the 1/16" GRP/VSP Tube Ferrule (Part #184929), and the 1/16" TFE Tube Ferrule (Part #177626) should be replaced when a proper seal cannot be obtained. ***Do not overtighten*** fittings in an attempt to maintain a proper seal. Inspect all Ferrules during regular maintenance procedures (i.e., Transfer Line Rinsing, Reaction Tube replacement), and replace as necessary.

Maintaining the Transfer Line

The Transfer Line between the Reactor and the Cell Assembly can become contaminated with use, resulting in peak tailing, loss of response, and/or baseline noise. In most instances this contamination can be removed by rinsing the Line with solvent (see "Transfer Line Rinsing"). If no improvement is observed, the Transfer Line should be replaced. Replace the Halogen Mode Transfer Line with approximately 10 inches of 1/16" x 0.020" I.D. TFE tubing (Part #165896). Replace the Nitrogen or Sulfur Mode Transfer Line with approximately 4 inches of 1/16" x 0.030" I.D. PEEK tubing (Part #197202).

Replacing Scrubber

Periodic replacement of the chemical Scrubber is required to maintain proper ELCD selectivity (for Nitrogen and Sulfur Modes, see Fig. 5.2). Common symptoms that indicate the need to replace the Scrubber include: Detector response to Halogens, and a high baseline and/or baseline noise. Replace the Scrubber as described in the following procedure.

NOTE: The Reactor should remain **hot** while this procedure is performed.

- Turn the solvent flow OFF.

- Remove the Scrubber Union from the Scrubber by loosening the Scrubber Tube Nut between the Scrubber and the Union (see Fig. 5.2). Inspect the TFE Tube Ferrule (Part #177626) and replace, if necessary.

- Remove the Scrubber by removing the Knurled Reactor Nut at the top of the Reactor. Examine the 1/16" TFE Ferrule (Part #156689) seated in the Reactor Nut and replace, if necessary.

- Install a new Scrubber by inserting the Knurled Reactor Nut and TFE Ferrule onto the Scrubber and ***finger-tightening*** the Reactor Nut onto the Reactor Union. See Fig. 5.2 for proper Scrubber orientation. Use a Nitrogen Scrubber (Part #170655) or a Sulfur Scrubber (Part #170580).

- Reinstall the Scrubber Union with the attached Transfer Line to the Scrubber. All fittings should be ***finger-tight.***

- Check for leaks, but do ***not*** use any liquid leak detectors around the Reactor Assembly.

Cell Removal

The cell may be cleaned when necessary to remove lodged particles or other residue accumulated on the electrodes. Signs of a contaminated Cell include poor response and/or high or noisy baseline. Remove the Cell according to the following procedure:

NOTE: This procedure requires Micro® all purpose cleaner (Part #259945)

- Turn the solvent flow OFF.

- Remove the Cell Enclosure Cover.

- Remove the Solvent Lines from the Cell by removing the 3 Thumb Nuts. (see Fig. 5.4).

- Remove the Cell by pulling it away from the two electrode posts.

NOTE: Do not take the Cell halves apart. Disassembling these parts will void the warranty on the Cell Assembly.

- Prepare a 2% Micro® solution in a 250 ml beaker by mixing one part Micro solution to 50 parts D.I. water. Place the Cell in the beaker containing the 2% solution, ensuring the Cell is completely immersed.

- Place the beaker in a sonicator of water and ultrasonically clean it for 30 minutes at 50°C.

- Remove the Cell and rinse thoroughly with D.I. water. This is best performed using a squirt bottle and flowing the D.I. water into the 3 ports of the Cell. Rinse one final time with methanol.

- If possible, blow dry the Cell with a clean gas supply. Complete the drying process by heating the Cell in the GC oven for at least 30 minutes at 75°C.

NOTE: A Cell that is not completely dry will cause a high baseline.

- Reinstall the Cell by carefully inserting the two electrode posts into the Cell.

- Connect the three Solvent Lines to the Cell (see Fig. 5.4). Do not overtighten the Thumb Nuts —***finger-tight*** will suffice.

- Check the gas flow rates (see "Set Gas Flows").

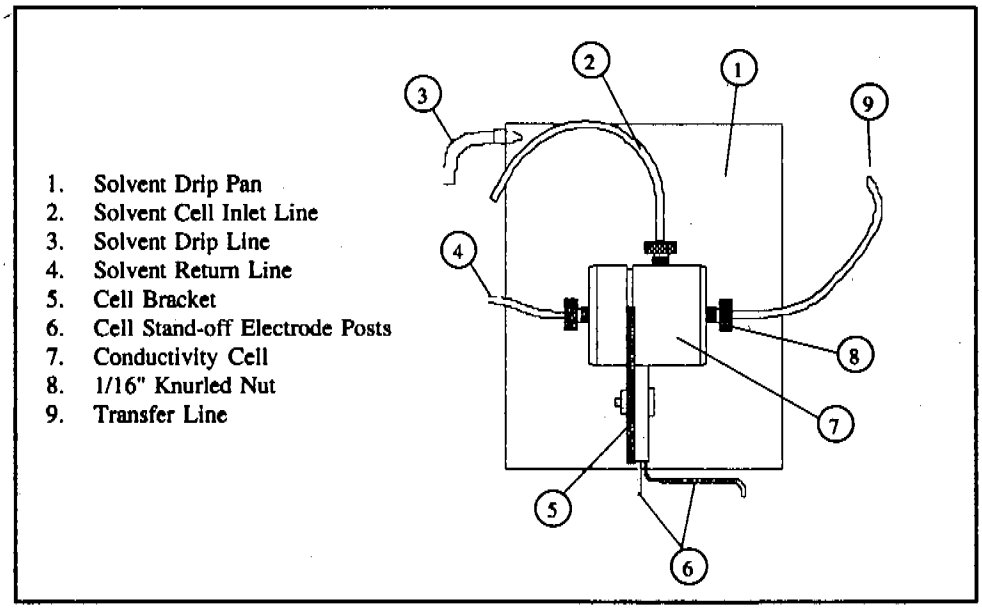


Figure 5.4. Cell Assembly

- Turn the solvent flow to ON and check for solvent leaks at the Cell. Check the Thumb Nuts for proper tightness if leaks occur, but do not overtighten. If a leak occurs at a properly tightened Nut, replace the TFE Ferrule (Part #156689) seated in the Nut.

- Replace the Cell Enclosure Cover.

Replacing Reactor Base Seal

If the Reactor Base Seal (Part #223933) becomes chipped, cracked, and/or deformed, replace it according to the following procedure:

- Turn the Solvent Pump and Reactor temperature OFF.

- Remove the Reactor (see "Replacing Reaction Tube").

CAUTION:
The nut is hot!

- For Tandem PID/ELCD, remove the Adapter Nut from the PID Manifold using either the 1/4" nut driver supplied in the Start-up Kit or a 1/4" open-ended wrench. Remove the seal from the Nut by cracking it with pliers.

- For Stand-Alone ELCD, firmly grasp the Reactor Base Seal with long-nose pliers and pull upward. The Seal may be removed more easily by cracking it with the pliers.

- Install a new Reactor Base Seal (Part #223933) onto Reactor Base (stand-alone ELCD) or the Adapter Nut (Tandem PID/ELCD) with the detail/line facing downward or towards the PID. Firmly press the Seal into place.

- Reinstall the Reactor with a new Reaction Tube (see "Replacing Reaction Tube").

Chapter 6
Troubleshooting

The following is a list of the most common troubles that can occur when using the ELCD, along with their most probable causes and corresponding corrective actions. Each symptom potentially may be caused by more than one problem. The probable causes of each symptom are listed in order of increasing seriousness. Each corrective action has been discussed earlier in this manual, under either an installation, operation, or maintenance procedure. Before using this guide, please become thoroughly familiar with the operation and maintenance information contained in previous chapters.

For each symptom, the last corrective action is to replace one of the three principal components: control module, reactor assembly, or cell assembly.

SYMPTOM	PROBABLE CAUSE	CORRECTIVE ACTION
No Response	Electronic device not connected properly	Review Installing the 5200 Detector Controller, Chapter 3. Correct any connection problems.
	No electrolyte flow	Repeat Set Solvent Flow in Chapter 4. Correct any blockage problems.
	No carrier or reaction gas flow	Repeat Install Column in Chapter 4. Correct any leak problems.
Low or Tailing Response	Incorrect reactor temperature	Reset to specified temperature.
	Reaction tube fouled	Repeat Replacing Reaction Tube in Chapter 5.
	Carrier inlet line to cell contaminated	Refer to Transfer Line Rinsing in Chapter 5.
	Column degraded	Replace column. Refer to Install Column in Chapter 4.
	Leak in GC injector system	Check injection septum for leak. Inspect and tighten other components as needed.
	Reactor failed	REPLACE REACTOR CORE
	Cell contamination	Repeat Cell Removal in Chapter 5.
High Baseline	Column bleed	Cool column while monitoring baseline to check.

SYMPTOM	PROBABLE CAUSE	CORRECTIVE ACTION
	Contaminated electrolyte	Repeat Refilling Solvent, and Replacing Resin Cartridge in Chapter 5.
	Cell wet or damaged	Dry cell in oven at 100° C for 1 hour, then retest.
Spikes in Baseline	Reaction tube not conditioned	Allow reaction tube to condition at 950° C for 10 minutes, then retest.
	Reaction tube fouled	Repeat Replacing Reaction Tube in Chapter 5.
	Particle eluting from column	Filter column effluent with 5µm frit.
	Incorrect carrier to reaction gas ratio	Check gas flow rates separately at exit of solvent return line. Refer to Set Gas Flows in Chapter 4.
Noisy Baseline	Incorrect electrolyte flow	Measure electrolyte flow per *Set Solvent Flow in* Chapter 4.
	Electronic noise	Ensure cell enclosure cover is in place.
Insufficient electrolyte flow	Clogged solvent filter	Replace resin cartridge. See Replacing Resin Cartridge in Chapter 5.

Chapter 7
Replacement Parts

Throughout this manual, the various components of the 5220 have been identified and described. This chapter is a listing of the order numbers for these components and for other replacement parts and support items. Replacement parts that OI Analytical considers expendable (XPND) are marked with an asterisk. Expendable components are ones that are to be replaced regularly or are easily broken or deformed.

PART NAME	PART #	U/M	XPND
Assy Blank	254490	ea	
Cable Signal out to Spades	215962	ea	
Cable Signal out to HP Int	229633	ea	
Cable Signal out to HP AIB	229641	ea	
Cable - Reactor Interface	231365	ea	
Cable - Cell & Reaction Interface	216010	ea	
Cartridge - Resin Hx Disposable 5220	213579	ea	*
Cartridge - Resin N Disposable 5220	230524	ea	*
Cartridge - Resin S Disposable 5220	225003	ea	*
Cell Assy - 5220 Hp 5890	234237	ea	
Ferrule - Br 1/16 Tube	223776	6 pk	*
Ferrule - GRP/VSP 1/32 x .5 mm Tube	231043	ea	*
Ferrule - GRP/VSP 1/32 x .8 mm Tube	231050	ea	*
Ferrule - GRP/VSP 1/16 Tube	216366	10 pk	*
Ferrule - TFE 1/16 Tube	196220	5 pk	*
Fitting - Union Assy 1/16 Reaction Top	226464	ea	
Fitting - Nut SS 1/16 Female Knurled	226357	ea	
Kit - Halogen Mode 5220	219402	ea	*
Kit - Nitrogen Mode 5220	224972	ea	*
Kit - Startup 5220 HP 5890	212563	ea	
Kit - Sulfur Mode 5220	224980	ea	*
Manual - Operation & Service - 5220	224923	ea	
Nutdriver 1/4 Hollow	223917	ea	
PCA - 5220 Cell Amplifier	259416	ea	
PCA - 5220 Cell Interface	223222	ea	
Reactor Assy - 5220	227496	ea	
Reactor - Core 5220	234187	ea	
Scrubber - Nitrogen Mode	170655	ea	*
Scrubber - Sulfur Mode	170580	ea	*
Scrubber Union	204735	ea	
Solvent Flow Assy 5220 HP 5890	224519	ea	
Solvent Pump Assy - 5220	216267	ea	
Solvent Reservoir	216507	ea	
Tube - Alumina Reaction	217203	3 pk	*
Tube - Ni Reaction 5220	260323	12 pk	*
Tubing - PEEK 1/16 x .030 I.D.	197202	ft	
Tubing - TFE 1/16 x .038 I.D.	234401	ft	*
Tubing - TFE 1/16 x .007 I.D.	272542	in	*
Tubing - TFE 1/16 x .020 I.D.	165896	in	*

NOTES