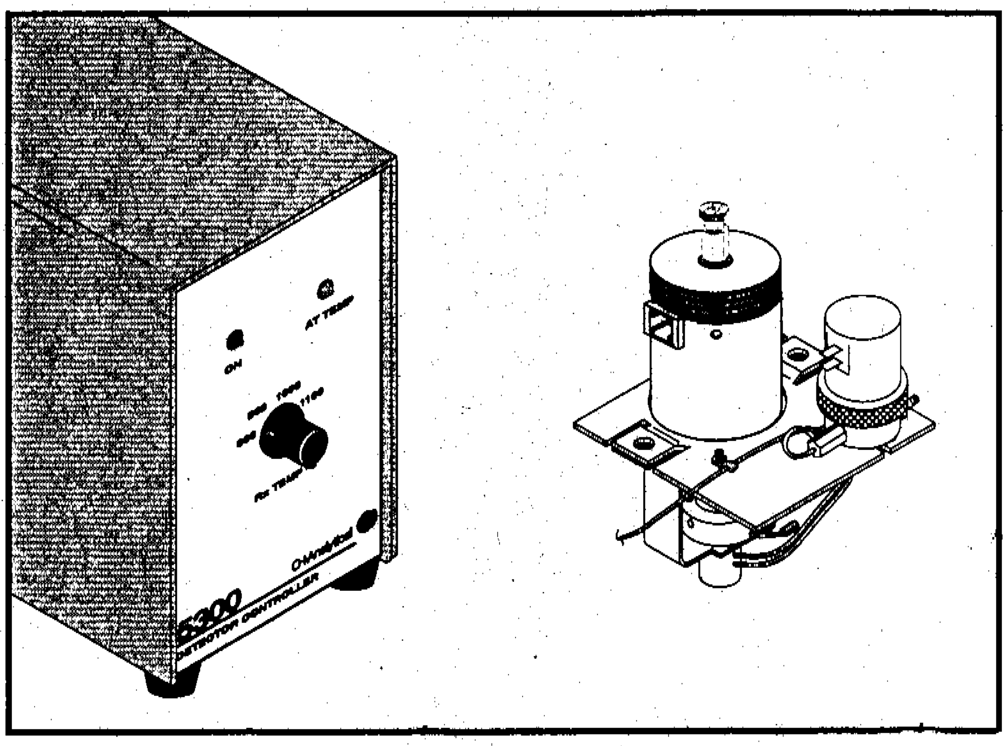
Model 5320 Electrolytic Conductivity Detector Operator's Manual—HP 5890 Series GC



O·I·Analytical

151 Graham Road · P.O. Box 9010 · College Station, Texas 77842-9010
Telephone (409) 690-1711 · FAX (409) 690-0440

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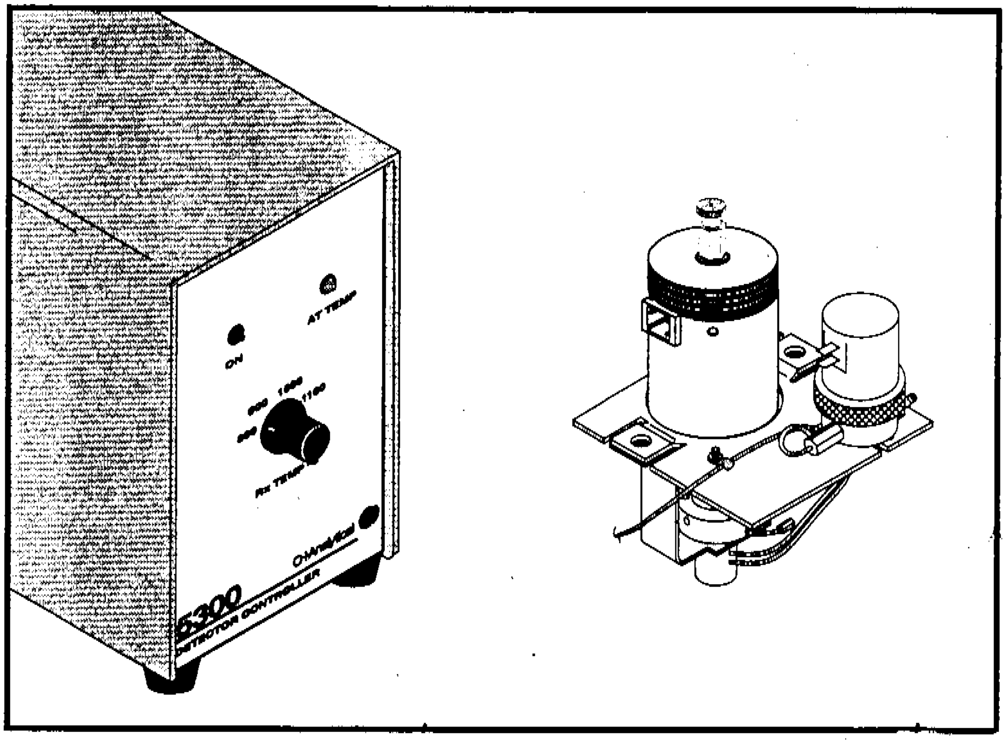

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OI Analytical's Model 5320 ELCD is protected under U.S. Patents #4,649,124 and #4,917,709.

OI Analytical Part #274969

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Limited Warranty

OI Analytical warrants each Model 5320 Electrolytic Conductivity Detector (ELCD) against defects in materials and workmanship under normal use and service for a period of ninety (90) days. Equipment installed by OI Analytical is warranted from the installation date; all other equipment is warranted from the ship date. If purchaser schedules or delays installation more than 90 days after delivery, then warranty period starts on the 91st day from date of shipment. This warranty extends only to the original purchaser. OI Analytical will, at its option, repair or replace equipment that proves to be defective during the warranty period, provided the equipment is returned to OI Analytical at the expense of the purchaser. Parts, labor, and return shipment to the customer shall be at the expense of OI Analytical. Parts used and labor performed during on-site warranty service requested by the purchaser shall be at the expense of OI Analytical.

Consumables, columns, and high temperature furnaces are warranted for 30 days (parts only) and are not available for coverage under extended warranties or service contracts.

Units purchased without on-site installation will be returned to the factory for repair. Units purchased with on-site installation will be repaired on-site at the discretion of OI Analytical.

This warranty shall not apply to defects originating from:

- Improper maintenance or operation by purchaser.
- Purchaser-supplied accessories or consumable.
- Modification or misuse by purchaser.
- Operation outside of the environmental and electrical products specifications.
- Improper or inadequate site preparation.
- Purchaser-induced contamination or leaks.

THE FOREGOING WARRANTY IS IN LIEU OF ALL OTHER WARRANTIES, EXPRESS OR IMPLIED, INCLUDING BUT NOT LIMITED TO ANY WARRANTY OF MERCHANTABILITY, FITNESS, OR ADEQUACY FOR ANY PARTICULAR PURPOSE OR USE. OI ANALYTICAL SHALL NOT BE LIABLE FOR ANY SPECIAL, INCIDENTAL, OR CONSEQUENTIAL DAMAGES, WHETHER IN CONTRACT, TORT, OR OTHERWISE.

Any service requests or questions should be directed to the Customer Service Department at 1-800-336-1911.

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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 5320 Electrolytic Conductivity Detector represents a true advance in detection technology. The Model 5320 has been specifically designed as a lower cost alternative to our Model 5220 ELCD aimed at the detection of halogenated compounds eluting from capillary GC columns. Major improvements have been made in many detector components, particularly the electronic systems.

Detector Design

The OI Analytical Model 5320 ELCD consists of three principal components (see Figure 1.1):

- Model 5300 Detector Controller
- Reactor Assembly
- Model 5320 Cell/Solvent Assembly

The Model 5300 Detector Controller houses reactor control and power supply electronics. It is functionally independent of the GC. The Model 5320 cell/solvent system houses the cell, conductivity amplifier, signal output connector, and solvent system. The reactor assembly houses the reactor, reaction tube, reactor base, and vent valve.

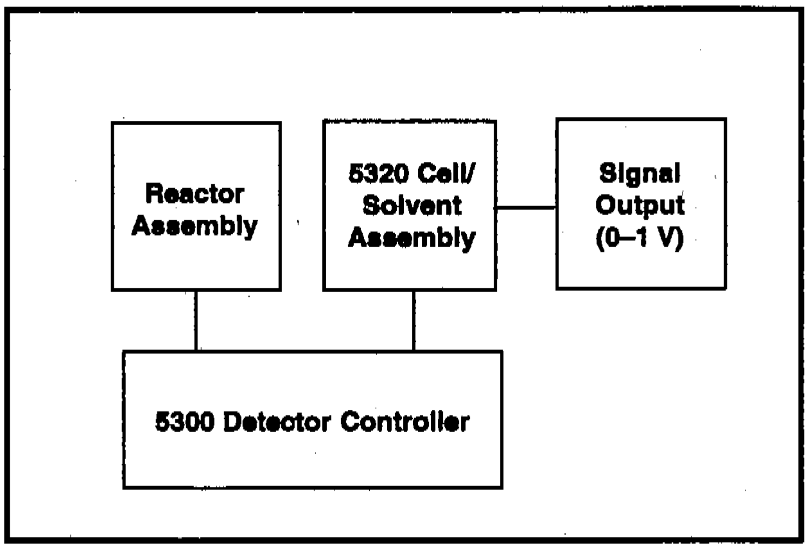

Figure 1.1. Model 5320 ELCD Principal Components

The three possible main operating modes for the Model 5320 ELCD are: Halogen (H), Sulfur (S), and Nitrogen (N). OI Analytical offers an operating kit for the Halogen Mode. Operating kits for Sulfur and Nitrogen Modes will be added as options at a later date. 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 the 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 0–1 V signal and output via a signal cable to an external data output device, which results in a chromatogram.

The vent valve (controlled by the GC's timed events) provides 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 adjustments are available on the cell assembly board.

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 the solution.

The Model 5320 ELCD's principle of response is based on converting the analyte to small, ionizable, inorganic molecules, and detecting 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 the 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.

Features

Operating Parameters for Selective Detection

Mode	Rx Temp (°C)	Rx Gas	Conductivity Mode Solvent	Scrubber
Halogen (H)	900 - 1100	H_2	100% ACS Reagent Grade n-propanol.	None
Sulfur (S)	800 - 1100	Air	100% ACS Reagent Grade methanol.	S-Mode
Nitrogen (N)	800 - 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 X-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

- Low maintenance cell with quick-disconnect attachments.
- Compact modular design.
- Directly interfaces to several GC makes and models.
- Improved design allows reaction tubes to be replaced quickly and easily.
- Direct interface to the OI Analytical Model 4430 or Model 5230 Photoionization Detector (PID) without transfer line (using single detector port).
- Minimal operator-interface or adjustments.
- 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 and solvent flow are analog-controlled.
- Simplified signal processing.
- Detector base and Model 5300 Detector Controller are interchangeable with the OI Analytical Model 5360 Halogen Specific Detector (XSD™).

- Control module incorporates quick-change, disposable deionizing cartridge and simplified solvent system.
- PID can be added to form integral dual-detector.
- Vent(s) may be turned on and/or off using the GC's timed events options.

Principal Applications

- EPA 601
- EPA 608
- EPA 611
- EPA 502.1
- EPA 502.2
- Fluorinated and Chlorinated Contaminates in Process Streams
- 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

- Controlled by GC timed events relay (24 V)

Dimensions (5300 Detector Controller)

- 8.25" H x 5.00" W x 12.0" D

Weight

- Controller: 8.4 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: 800° to 1100°C (in 100°C increments)
- Stability: ±1°C

Solvent Flow

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

Detector Output

- 1 V full scale analog voltage

Gas Requirements

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

Power Requirements

- 90–260 VAC (±10%)/47–63Hz, 200 W

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

Safety Information

The Model 5320 ELCD has been designed in accordance with recognized safety standards and for use indoors. Using the instrument in a manner not specified by the manufacturer may impair the instrument's safety protection. Whenever the safety protection of the Model 5320 ELCD has been compromised, disconnect the instrument from all power sources and secure the instrument against unintended operation.

Operator Precautions

For operator safety, pay attention to *WARNING* and *CAUTION* statements throughout the manual.

- A *WARNING* indicates a condition or possible situation that could result in physical injury to the operator.
- A *CAUTION* indicates a condition or possible situation that could damage or destroy the product or the operator's work.

Warnings and precautions in this manual or on the instrument must be followed during operation, service, and repair of the instrument. Failure to follow these warnings and precautions violates the safety design standards and intended use of the instrument. OI Analytical will not be liable for the operator's failure to comply with these warnings and precautions.

The Model 5320 ELCD must be connected to the AC power supply mains through a three-conductor power cord with the third wire firmly connected to an electrical ground at the power outlet. Any interruption of the grounding conductor or disconnection of the protective earth terminal could cause a shock resulting in personal injury.

General Precautions

- Disconnect the AC power cord before removing any covers.
- Replace or repair faulty or frayed insulation on power cords.
- Perform periodic leak checks on supply lines, fittings, and pneumatic plumbing.
- Arrange gas lines so that they can not become kinked, punctured, or otherwise damaged, and will not interfere with foot traffic.
- Turn off the main power switch and disconnect the main power cord before using a liquid solution to locate leaks.
- Do not restrict airflow on the back and/or bottom of the unit.
- Wear safety glasses to prevent possible eye injury.
- Do not replace blown fuses inside the detector controller. Only trained service personnel should access the interior of the detector controller.
- Do not perform unauthorized modifications or substitute parts that are not OI Analytical original instrument parts. Any unauthorized modifications or substitutions will void the warranty.
- Verify that all heated areas have cooled before handling or wear adequate hand protection to prevent burns.

Compressed Gas Cylinders Precautions

- Compressed gases should be stored and handled strictly in accordance with relevant safety codes.
- Fasten all cylinders securely to an immovable structure or permanent wall.
- Store or move cylinders only in a vertical position. Do not move or transport cylinders with regulators attached.
- Use only approved regulators and tubing connections.
- Connect cylinders to instruments with pressure ratings that are significantly greater than the highest outlet pressure from the regulator.

Safety Symbols

The following symbols may be located on the instrument:

See accompanying instruction for more information.

Indicates a hot surface.

Indicates hazardous voltages.

Indicates earth (ground) terminal.

Notes

Chapter 2
Description of Components

Principal Components

Model 5300 Detector Controller provides control for the reactor temperature and power to both the reactor and cell assembly. The Model 5300 can control one Model 5320 ELCD or one Model 5360 XSD™. Electrical components include the power supply, reactor temperature controller, ON power and AT TEMP LEDs, and temperature set switch.

Model 5325 Cell/Solvent Assembly includes the conductivity cell, its enclosure and mounting hardware, its associated fluid-flow and electrical lines, the solvent pump and reservoir, and the conductivity amplifier. This assembly houses the "sensor" portion of the detector—the conductivity cell. Conversion of the signal from the conductivity cell to a usable output occurs on the cell amplifier board in the cell assembly. The conductivity amplifier generates a 0–1 V signal, which is output to a data handling device via a signal cable. The cell amplifier board contains all of the devices (switches and pot adjustments) necessary to set the detector's operating parameters (excluding temperature).

Model 5320 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.

Model 5300 Detector Controller - Front View

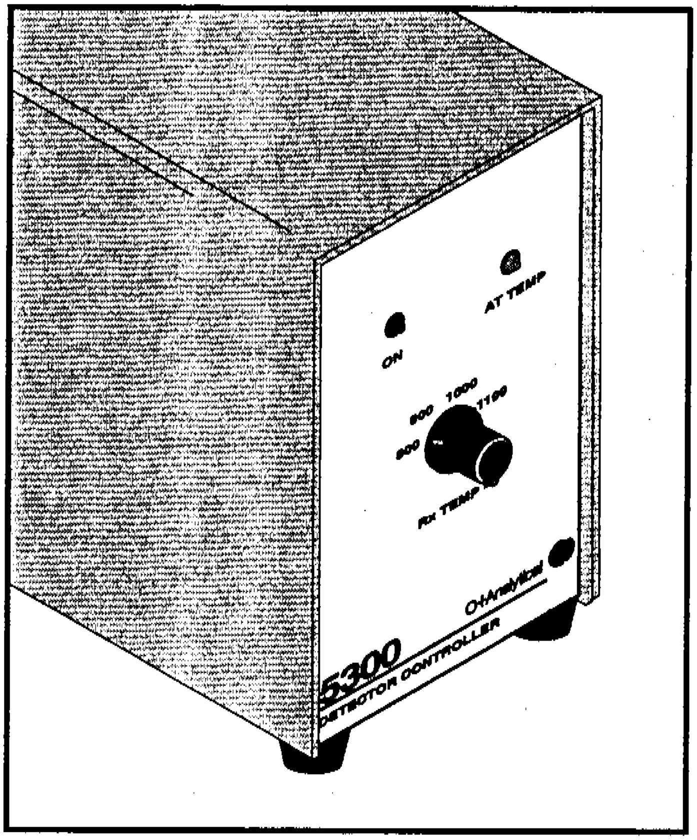

Figure 2.1. Detector Controller - Front View

AT TEMP LED will turn on when the temperature in the reactor reaches the temperature that was selected. This LED will go off when the reactor is not at the specified temperature or when the power is off.

ON LED indicates the power status. The LED will be on when the power is turned on to the controller.

Temperature Select Switch (Rx TEMP °C) selects the reactor temperature between 800°–1100°C in 100°C increments. Reactor temperature calibration is factory set.

Model 5300 Detector Controller - Back View

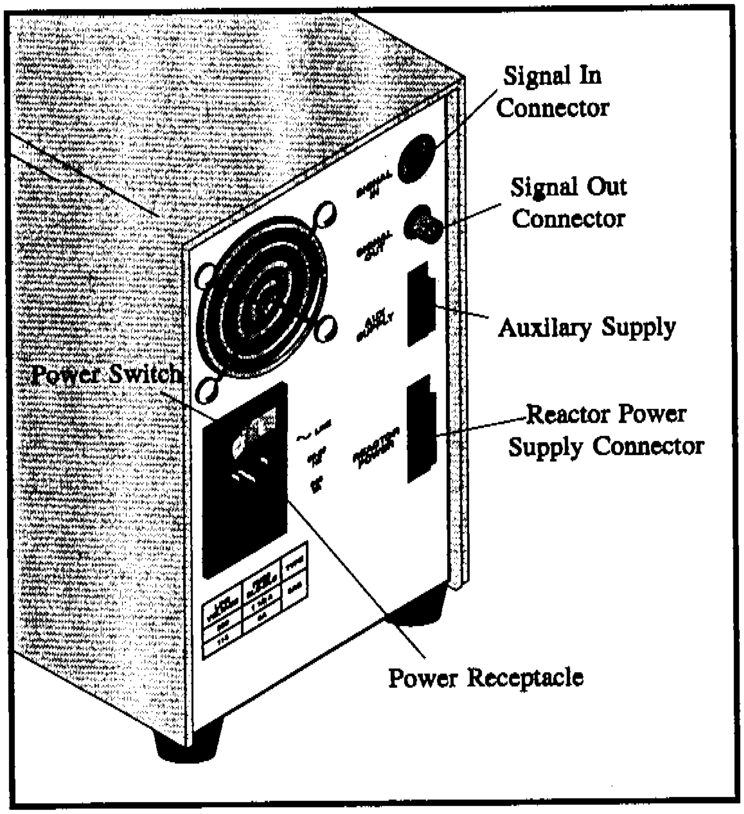

Figure 2.2. Detector Controller - Back View

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

Power Switch (rocker switch) turns the reactor power, bias voltage, electrometer power, and cell amplifier board power on/off. Power status is indicated by the ON LED on the front of the controller.

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

Reactor Power Supply Connector (3-pin Molex connector) joins the reactor power cable to supply power to the reactor.

Signal In Connector (6-pin connector) joins with the signal probe cable in the Model 5360 XSD. This connector is not used with the Model 5320 ELCD.

Signal Out Connector (BNC connector) provides a signal output voltage with a nominal range of 0 to 1 volt for the Model 5360 XSD. This connector is not used with the Model 5320 ELCD.

Model 5325 Cell/Solvent Assembly - Top View

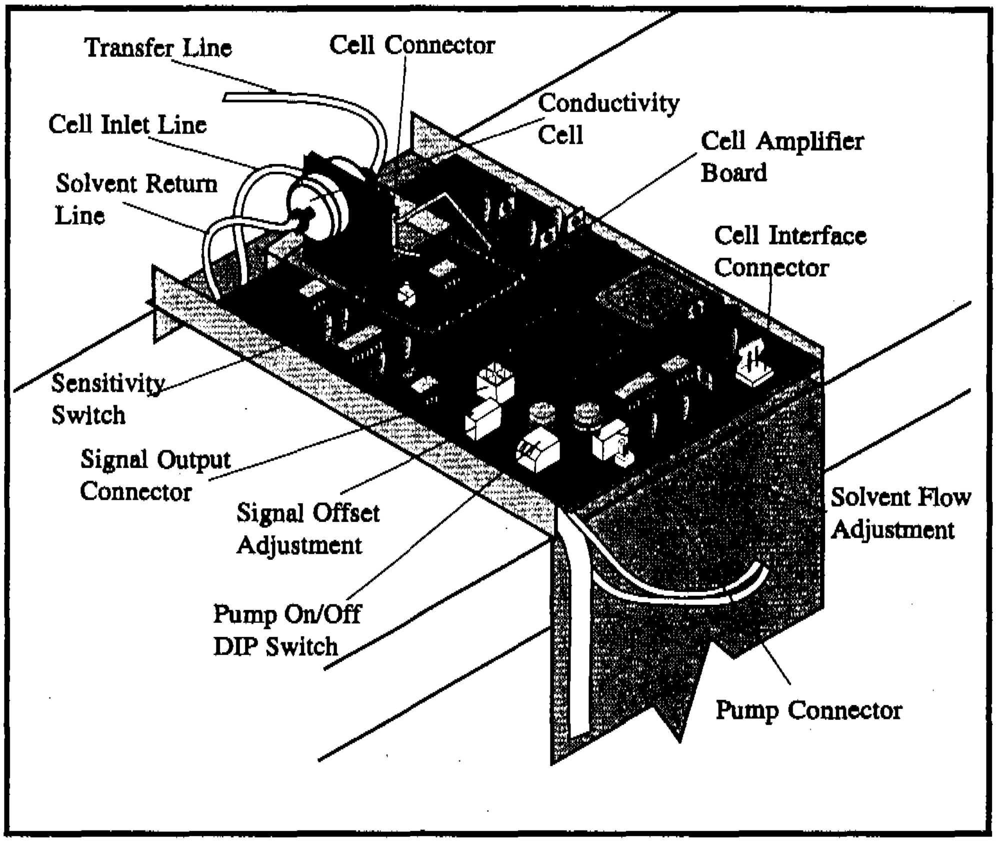

Figure 2.3. Cell/Solvent Assembly - Top View

Cell Amplifier Board controls most of the detector's operation, power supply, and signal processing. Many detector parameters are controlled or adjusted at various locations on this board.

Cell Connector (2-pronged connector on U-bracket) allows easy installation of the conductivity cell. (See close-up in Figure 5.4.)

Cell Inlet Line carries solvent from the solvent block to the conductivity cell (uses reverse ferrule and knurled nut).

Cell Interface Connector (2-pin connector) joins with the cell interface cable to the Model 5300 Detector Controller supplying power to the cell/solvent assembly.

Chassis Ground (not shown) is the ground connection to the GC.

Pump Connector (2-pin connector) connects the cable that supplies power to the solvent pump to the cell/solvent assembly.

Sensitivity Switch switches the detector between high and low sensitivity.

Signal Offset Adjustment increases or decreases the signal output from the detector (usually used if changing modes).

Signal Output Connector (4-pin connector) provides a signal output voltage with a nominal range of 0 to 1 volt. The detector output cable (ordered separately) provides the detector signal to an HP AIB board or other data handling device. Other cables are available when connecting to other makes or models of data handling devices.

Solvent Flow Adjustment sets and adjusts the solvent flow going to the conductivity cell.

Solvent Return Line transfers waste solvent and gas to the solvent reservoir (uses reverse ferrule and knurled nut).

Transfer Line transfers reaction products from the reactor to the cell (uses reverse ferrule and knurled nut).

Model 5325 Cell/Solvent Assembly - Side View

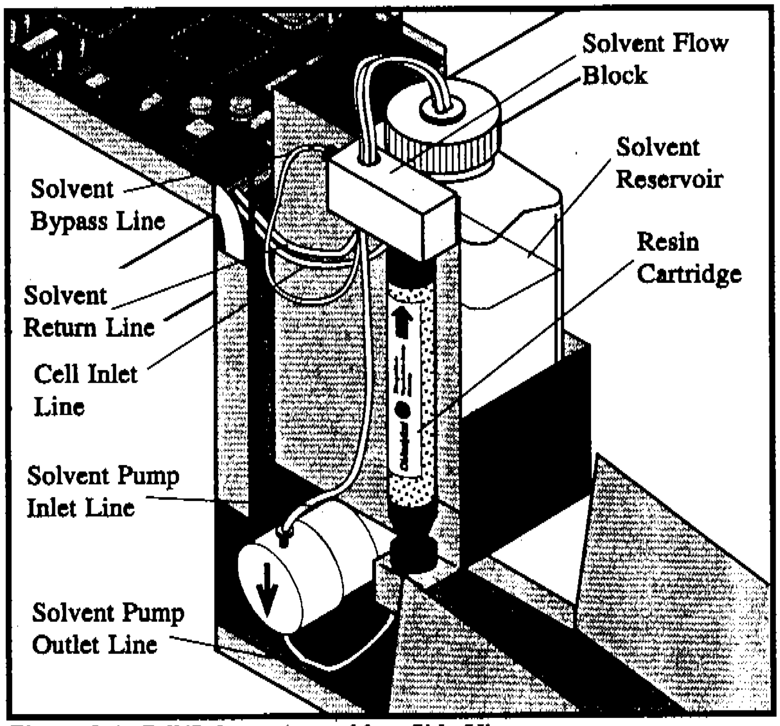

Figure 2.4. Cell/Solvent Assembly - Side View

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

Solvent Bypass Line carries excess solvent from the solvent flow block to the solvent reservoir. Only a small portion of the solvent pumped is required at the cell.

Solvent Flow Block accepts solvent from the solvent pump and resin cartridge and splits it between the solvent bypass and cell inlet line. All connections are made with a knurled nut and a double-sided PEEK ferrule.

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.

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

Model 5320 Reactor Assembly

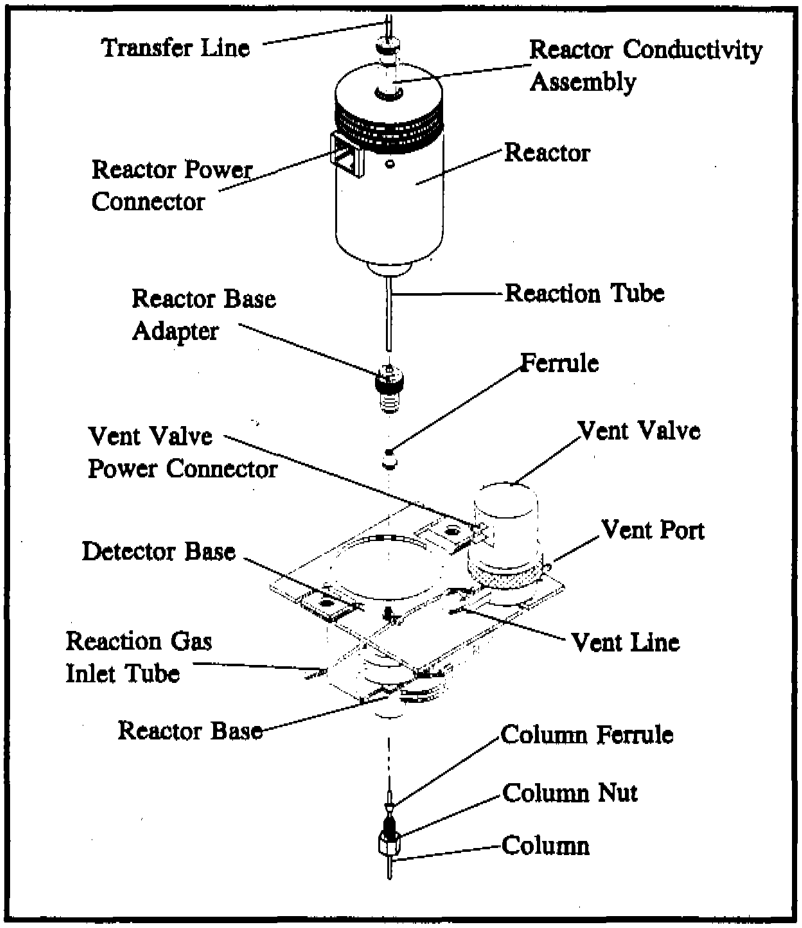

Figure 2.5. Reactor Assembly

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 reactor base.

Reaction Tube 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 specially treated nickel; for the Sulfur mode, an alumina tube is standard. Other materials may be used for specific applications.

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 provides a connection to the GC column, allows reaction gas (hydrogen or air) to introduced, allows the vent line to connect to the vent valve, and supports the reactor. This base mounts the detector securely into the detector port of the GC. The base is specifically designed for each particular GC model.

Reactor Conductivity Assembly (not shown) 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. (See detail in Figure 3.2.)

Reactor Power Connector (4-pin connector) mates with the reactor power cable and makes the power connection between the Model 5300 Detector Controller and the reactor heating filament.

Solenoid Valve Cable (not shown) connects to GC timed events relays (or cable) to control vent valve functioning.

Vent Valve, when closed (de-energized), allows all reaction gas to enter the detector base and flow through the reaction tube. When open (energized), the vent valve allows most of the GC effluent and some reaction gas to pass through the lower vent line and out the valve, instead of through the reaction tube. It is controlled and programmed using the GC timed events control.

Vent Line connects to the vent valve so that unwanted GC effluents pass through the valve instead of through the reactor when the vent valve is open.

Vent Port (outlet fitting) is the exit port for the flow of reaction (and column effluent) gas through the reactor when the vent is open.

Vent Valve Power Connector joins with the vent cable. It makes the connection between the vent valve and the vent cable.

Notes

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.

A signal cable for connecting the signal output of the detector to a data handling device is required. (Electrolyte is not supplied: electrolytes are specified in Table 4.1 of Chapter 4. All alcohol used for electrolyte should be ACS Reagent Grade quality.)

Stand-Alone 5320 ELCD or 5322 Dual ELCD Installation onto an HP 5890

Preparing the GC

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

Installing the Reactor Base and Reactor

- Insert the detector bracket with the reactor base into the appropriate detector port. (Do not remove the top plastic plug from the top of the reactor base until ready to install the reaction tube.)
- Align the holes of the detector base with the holes in the top of the GC, and screw the detector base onto the GC.
- Route the reaction gas line along the top of the GC using the existing tubing guides.
- Attach the gas line from the reactor base to the port of the gas flow module on the left side of the GC. If a gas flow module is not installed, see "Installing the Gas Flow Module" in this chapter.
- Install the reaction tube and brass ferrule (Part #223776) into the reactor base by tightening the reactor base adapter (Part #223743) with a 1/4" nut driver (supplied in start-up kit). See Figure 3.1. Verify that the reaction tube is fully seated into the reactor base.

CAUTION: *Verify that the reaction tube is in place. This location is the most common place that a leak is found.*

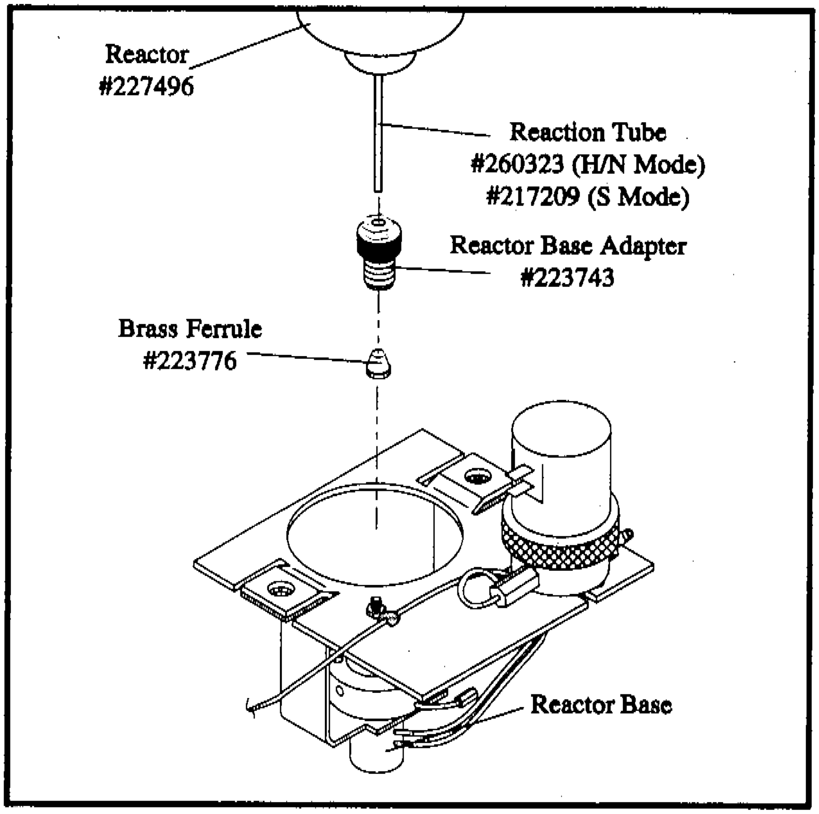

Figure 3.1. Model 5320 ELCD

- Slide the reactor over the reaction tube.
- Turn the reactor while slightly pressing downward, to ensure proper seating of the reactor onto the reactor base.
- Slide and properly seat the reactor top fitting (Part #227462), GRP/VSP ferrule (Part #216366), and reactor union (Part #234021) over the reaction tube and slightly tighten down. (see Figure 3.2).
- Attach the reactor power cable (Part #245506) to the reactor power connector on the detector, and route the cable to the back of the GC.
- Connect the other end of the reactor power cable to the reactor power supply connector on the back of the Model 5300 Detector Controller.
- Plug the vent valve cable (Part #214890) into the vent valve (verify that the vent is screwed down securely). Route the cable to the back of the GC and into the right side of the GC. Attach the pins on the end of the cable into the appropriate connectors on the HP 5890 main board (back top corner).
- Remove the bottom plastic plug from the detector base.
- Open the GC oven door to install the column. Slide the column through the nut (Part #223057) and the appropriate GRP/VSP tube ferrule. Note the proper orientation of the ferrule, with the tapered end toward the nut (see Figure 3.3).

- Gently push the column into the reactor base as far as it will go (only 1–2 mm beyond the back of the ferrule). Using a 1/4" wrench, tighten the nut approximately 1/4-turn past snug while exerting a slight upward pressure on the column, then pull down lightly to verify that the column is not broken. See "Installing the Column" in this chapter for further details.

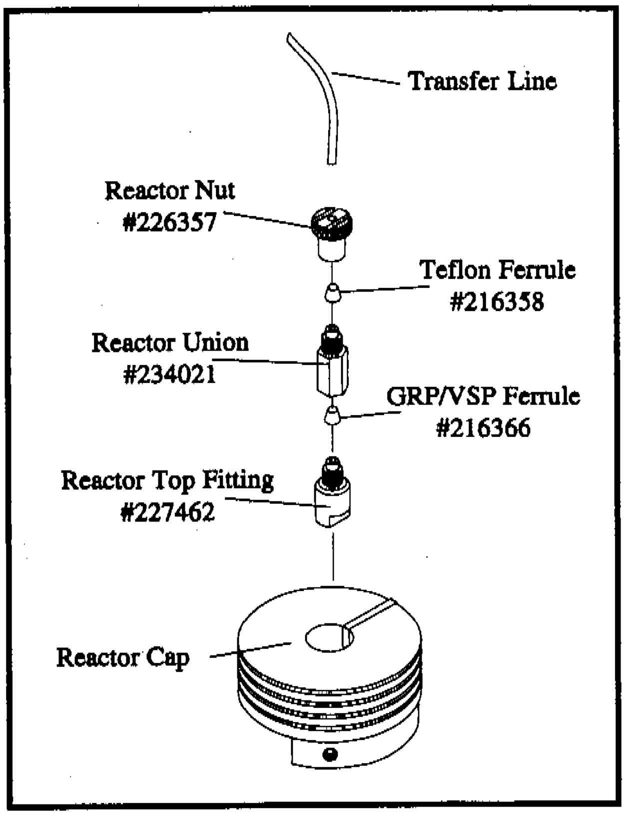

Figure 3.2. Reactor Conductivity Assembly

- If using a remote start cable, plug it in and route the cable along the top, to the back of the GC. Extend the cable out of the back of the GC so that the end of the cable will be available after the cover is put back in place.

- Replace the upper right and left side GC cover. If installing a gas flow module, do not replace the left side GC cover.

Installing the Cell/Solvent Assembly

CAUTION: Failure to attach the grounding strap will lead to excessive noise in the ELCD signal output.

- With the upper right GC cover put back in place, hang the cell/solvent assembly over the right side of the GC so that the tab on the cell/solvent assembly locks under the GC cover.

- Attach the grounding strap from the cell/solvent assembly to the top of the GC with the supplied screw. This will properly ground the Model 5320.

- Remove the top cover of the cell/solvent assembly by gently pulling upward.

- Connect the other end of the ground strap to the grounding screw on the solvent assembly if it is not already attached.

- Plug the detector signal cable (appropriate cable is ordered separately from the detector) into the 4-pin signal output connector on the cell amplifier board. Connect the other end of the cable to the particular data handling device to be used with the Model 5320 ELCD. When using an HP Analog Input Board (AIB), this cable will plug into the AIB on the top right side of the GC. Route this cable along the top, to the back of the GC (see Figure 2.3).

CAUTION: Overtightening the nuts onto the conductivity cell may result in insufficient solvent flow through the cell.

CAUTION: 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 detector base, and irreversibly fouling the reaction tube.

- Plug the cell interface cable (Part #247007) to the cell interface connector on the cell amplifier board (see Figure 2.3).
- Route the cell interface cable through the cable fastner and out the back of the cell solvent assembly.
- Route the cell interface cable to the back of the GC.
- Make a transfer line by cutting a **minimum** length of 1/16" x .20 I.D. Teflon® tubing that will extend from the top of the reactor to the conductivity cell located on the cell amplifier board.
- Connect one end of the transfer line to the conductivity cell by sliding the Telfon transfer line through the 1/16" male knurled nut (Part #226357) and 1/16" Teflon ferrule (Part #272443).
- Finger-tighten the 1/16" male knurled nut and transfer line into the conductivity cell. Do not overtighten (see Figure 5.4).
- Connect the other end of the transfer line to the top of the reactor by sliding the tubing through the 1/16" reactor nut (Part #226357) and Teflon ferrule (Part #216358). Note the direction of the ferrule in Figure 3.2.
- Finger-tighten the 1/16" reactor nut and transfer line onto the top of the reactor.
- Open the solvent enclosure by pulling outward on the side cover of the cell/ solvent assembly.
- Remove the solvent reservoir lid.
- Fill the solvent reservoir with the appropriate solvent for the application (see Table 4.1).
- Replace the solvent reservoir lid.

Installing the Column

The Model 5320 ELCD base is optimized for 0.53 mm and smaller I.D. capillary columns. To install the column into the detector base:

- Remove the 1/16" nut from the reactor base assembly or the start-up kit supplied with the Model 5320 ELCD.
- Slide the column nut onto the end of the capillary column.
- Slide a 1/16" GRP/VSP ferrule onto the column (with the tapered end facing toward the column nut) (see Figure 3.3). See Table 3.1 for the appropriate ferrule.

Base	Column	Ferrule I.D	Material I.D	Part #
1/16	0.53	0.8 mm	GRP/VSP	#196105
1/16	0.32	0.5 mm	GRP/VSP	#196113
1/16	< 0.32	0.4 mm	GRP/VSP	#208330

Table 3.1. Ferrule Specifications

- Cut 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 reactor base while finger-tightening the nut.

- Gently push the column into the reactor base as far as it will go (only 1–2 mm beyond the back of the ferrule). Using a 1/4" wrench, tighten the nut approximately 1/4-turn past snug while exerting a slight upward pressure on the column.

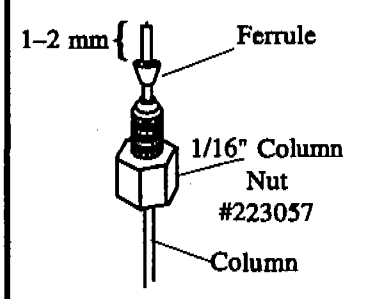

Figure 3.3. Capillary Column

- Leak-check the connection.

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 for an HP 5890 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 Figure 3.4).

- Slip the gas flow module into place on the back side of the front left panel as shown in Figure 3.4. The module fits over the two standoff posts and is fastened with the M4 x 45 mm mounting screw in the center location (**do not overtighten 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. Retighten the mounting screw and recheck the valve knob for freedom of movement.

- 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 the gas supply lines to the solvent flow block by using the manifold plate (Part #197772) and manifold O-ring (Part #185116) as in Figure 3.4.

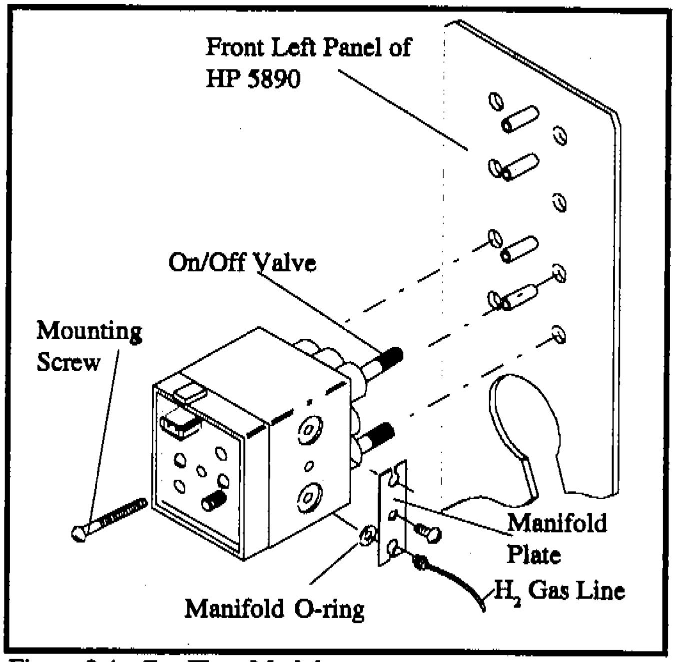

Figure 3.4. Gas Flow Module

Installing the Model 5300 Detector Controller

- Set the Model 5300 Detector Controller in place (preferably to the right side of the GC).

- Plug the power cable into the power receptacle on the back of the Model 5300 Detector Controller.

- Turn the power switch off.

- Plug the power cable into a standard 110 VAC power outlet.

- Plug the cell interface cable from the cell amplifier board into the auxiliary supply connector (labeled AUX SUPPLY) on the back of Model 5300 Detector Controller.

- Plug the reactor power cable into the reactor power supply connector (labeled REACTOR POWER) on the back of the Model 5300 Detector Controller.

Chapter 4
Operation

This chapter discusses the operation of the detector for analyzing samples. The Model 5320 ELCD can be operated in the Halogen, Sulfur, or Nitrogen Mode.

Setting the Gas Flows

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

Gas flows are set by adjusting the needle valve at the center of the larger on/off valve located on the gas flow module. Use a small screwdriver to adjust the needle valves; the valves open **counterclockwise**.

To adjust the gas flows, follow these procedures:

- Verify that the solvent flow is off.
- Remove the solvent return line from the solvent reservoir and attach a flowmeter to its end.
- Verify that the vent valve is closed.
- Set the column carrier gas flow to the desired rate.
- Open the reaction gas on/off valve at the gas flow module.
- For ELCD stand-alone operation, adjust the inner needle valve until the carrier-plus-hydrogen reaction gas flow equals 130 mL/min.
- For tandem PID/ELCD operation, open the makeup on/off valve at the gas flow module. Adjust the inner needle valve until the makeup gas flow equals 10–15 mL/min. Open the sweep gas on/off valve and adjust the lamp sweep gas flow until the column-plus-makeup-plus-sweep gas flow equals 145 (±5) mL/min.

CAUTION: Never adjust the needle valves to the point of complete shut-off to avoid inner-seal damage.

Note: In tandem operation, 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 the connections; **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.

Filling the Solvent Reservoir

The electrolyte in the solvent reservoir will slowly evaporate and must be refilled accordingly. Top-off the reservoir with solvent every 300 hours. Check the reservoir at least on a weekly basis.

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 4.1. 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 the solvent pump to be unable 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 4.1. The solvent reservoir holds 500 mL of electrolyte. Refill the reservoir routinely (see Chapter 5, "Maintenance").

Setting the Solvent Flow

Set the solvent flow according to the following procedure:

- Remove the solvent return line from the solvent reservoir, and insert the end into a small vial or container suitable for collecting solvent.

- Turn the solvent pump on using the pump on/off DIP switches located on the cell amplifier board (see Figure 2.3) Pushing the DIP switches up turns the pump on.

Note: If the solvent pump has been sitting idle for an extended period of time, prime the pump by setting the flow to a maximum setting. If the pump fails to prime, removing the exit line or pull the pump off the solvent assembly and invert the orientation of the pump.

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

- Allow the pump to stabilize for 5–10 minutes.

- Collect solvent in the small vial or container for 3–5 minutes.

- Measure the solvent by using a microliter syringe and determine the microliter per minute rate.

- Adjust the solvent flow until the flow rate is within the range given in Table 4.2.

- The solvent flow rate is adjusted using the solvent flow adjustment pot on the cell amplifier board (see Figure 2.3). Clockwise adjustment increases the solvent flow; counterclockwise decreases the solvent flow.

Mode	Rate
Halogen	25-40 µL/min
Nitrogen	40-80 µL/min
Sulfur	20-40 µL/min

Table 4.2. Solvent Flow Rates

- 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 conductivity cell.

- Check for solvent leaks.

The solvent flow rates given in Table 4.2 ensure reliable ELCD response. Generally, lower solvent flow rates increase detector response but also increase baseline noise. Using Table 4.2 as a guide, adjust the solvent flow rate to maximize the signal-to-noise ratio for any specific application.

The detector response is not dependent on exact repeatability of the electrolyte flow rate. Periodically, check the actual solvent flow rate by collecting solvent from the solvent return line.

Note: If the flow drops greatly, the resin cartridge may need to be replaced due to filter blockage or a solvent line may be overtightened.

Setting the GC Settings

CAUTION: Do not condition a new column while it is attached to the detector base.

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 Model 5320 ELCD base "tracks" or follows at approximately 30°C above the GC oven temperature. This feature minimizes column bleed.

Setting the Model 5320 ELCD Set Points

Most of the Model 5320 ELCD set points are controlled through adjustments made on the cell amplifier board. Use Table 4.3 as a guide to Model 5320 ELCD operation parameters. (Adjustments are shown in Figure 2.3.)

Control	Adjustment Location/Setting
Sensitivity Switch	Set switch to appropriate sensitivity
Signal (Zero) Offset	Determined by background noise and offset (pot adjustment on cell amplifier board)
Reactor Temperature	Set by front panel switch on Model 5300 Detector Controller
Rx Temperature Range	800°–1100°C
Halogen Mode	900°–1000°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	Controlled by GC timed events
Solvent On/Off	On/Off DIP switch on cell amplifier board
Solvent Flow	Pot adjustment on cell amplifier board

Table 4.3. Model 5320 Operation Parameters

Use the signal offset adjustment to adjust (increase or decrease) the signal output level on the Model 5320 ELCD. Adjusting this pot clockwise increases the signal offset; counterclockwise decreases the signal offset.

The sensitivity switch can be set to HI for pesticides analysis or LO for purge-and-trap analysis.

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 Model 5320 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.

- Use the highest purity gases available (99.999%) for the best signal-to-noise ratio. Proper gas purity and conditioning is crucial for successful ELCD operation.
- Replace the reaction tube, resin, and solvent lines when switching operating modes.
- If the resin is replaced, remove all solvent from the reservoir and refill it with new solvent.

Model 5300 Detector Controller Operation

The Model 5300 Detector Controller can control one Model 5320 ELCD.

- Set the temperature adjust switch to the appropriate temperature set point for the particular application to be run.
- Turn on the power to the Model 5300 Detector Controller. The ON (power) LED on the front of the Model 5300 Detector Controller will illuminate. Once the reactor reaches the set operational temperature, the AT TEMP LED will illuminate.

Notes

Chapter 5
Maintenance

Chapter 5 describes the scheduled and nonroutine maintenance of the detector.

Scheduled Maintenance

For the most reliable performance of the Model 5320 ELCD 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.

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

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.

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 the solvent pump to be unable 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 one of the following solvents every 300 hours (see Table 5.2). Check the reservoir at least on a weekly basis.

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 the resin cartridge 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 release 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.
- Dispose of any solvent remaining in the solvent reservoir. The solvent must be replaced when changing the resin cartridge
- Refill the solvent reservoir with new solvent. Refer to "Filling Solvent Reservoir" in this chapter, for proper solvent selection.
- Adjust the solvent flow adjustment on the cell amplifier board 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.
- 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 the Halogen Mode, unscrew the knurled reactor nut (Part #226357) at the top of the reactor (see Figure 5.1).
- For the Nitrogen/Sulfur Modes, disconnect the PEEK tubing (Part #197202) at the scrubber union (Part #204735) by loosening the scrubber tube nut (Part #204735) (see Figure 5.2).

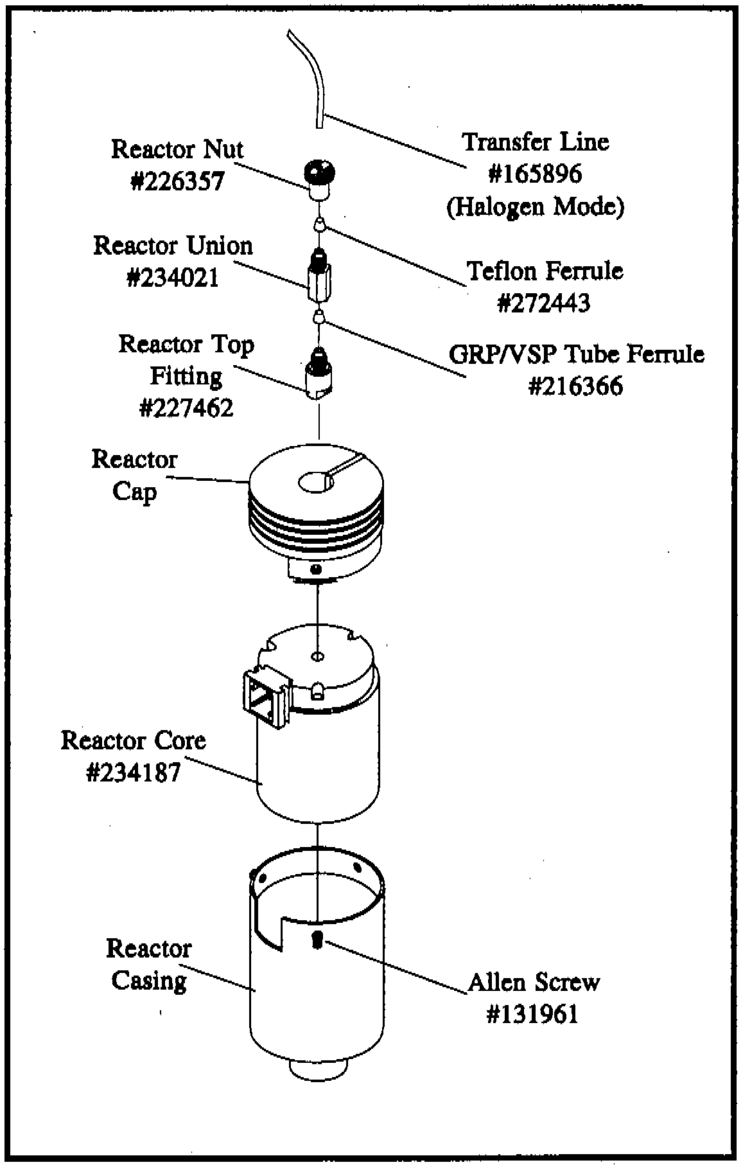

Figure 5.1. Reactor Assembly

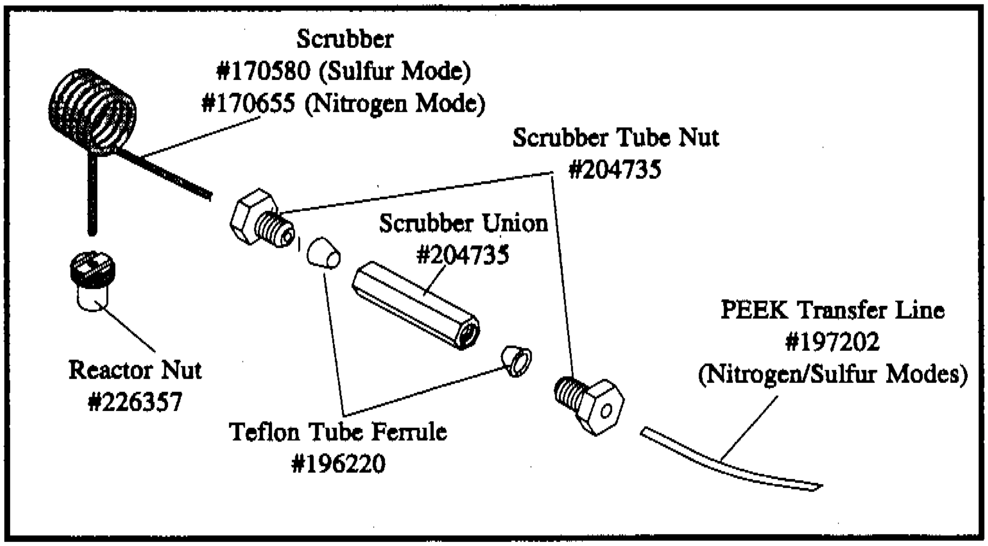

Figure 5.2. Scrubber Assembly

- With the solvent flow on, allow the transfer line to sag to an open area on the top of the GC, 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 conductivity cell, allowing the excess solvent to flow back into the conductivity cell.

- 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 Teflon ferrule used to seal the transfer line. Replace if necessary. Use the Teflon ferrule (Part #272443) for Halogen Mode or the Teflon tube ferrule (Part #196220) 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. Teflon 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).

Nonscheduled Maintenance

Replacing Reaction Tube

To replace the reaction tube, follow the steps listed below:

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

- Turn off the power supply to the Model 5300 Detector Controller.

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

- Turn off the solvent flow using the DIP switch on the cell amplifier board.

- Turn off the reactor gas supply.

- Disconnect the transfer line by removing the knurled reactor nut (Part #272443) (Halogen Mode) or the scrubber tube nut (Part #196220) (Nitrogen/ Sulfur Modes) at the scrubber union (see Figures 5.1 and 5.2).

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 (Part #226357) (Nitrogen/ Sulfur Modes).

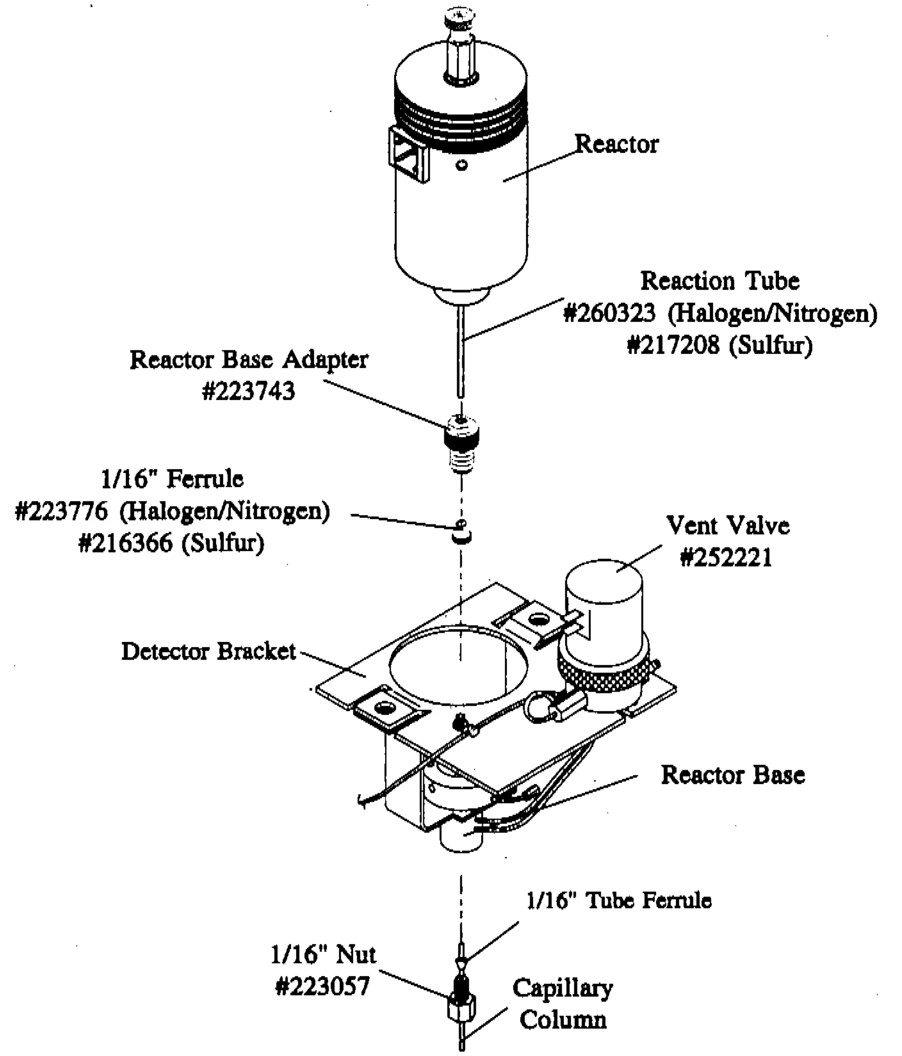

Figure 5.3. Reactor Base Assembly

- Remove the reactor union (Part #234021), GRP/VSP tube ferrule (Part #216366), and reactor top fitting (Part #227462) from the top of the reactor (see Figure 5.3).

- Slide the reactor off the reaction tube **(CAUTION: reactor may still be hot).**

- Remove the reaction tube using a 1/4" socket wrench (supplied in start-up kit) by sliding the wrench over the reaction tube and reactor base nut, and turning counterclockwise.

- Slide a new 1/16" ferrule, then the reactor base adapter over a new reaction tube. Use a nickel reaction tube (Part #260323) and a 1/16" brass ferrule (Part #223776) for the Halogen/Nitrogen Modes or an alumina reaction tube (Part #217208) and a 1/16" GRP/VSP ferrule (Part #216366) for the Sulfur Mode (see Figure 3.3).

- 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.

- 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 #216366) at the top of the reactor. Do not attach the transfer line to the reactor during reaction tube conditioning.

- Turn the power on at the Model 5300 Detector Controller and set the temperature to 900°C for approximately 10 minutes to "condition" the reaction tube.

- After conditioning the reaction tube, properly seat the top fitting onto the top of the 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 overtightened reactor nut. If necessary, trim the end of the transfer line to ensure a clean, straight cut. Examine the 1/16" Teflon ferrule (Part #272443) 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" Teflon ferrule (Part #272443). See Figure 5.2 for the proper scrubber orientation. The reactor nut needs to be only **finger-tight**. Examine the Teflon ferrule and replace, if necessary.

- For Nitrogen/Sulfur Modes, attach the PEEK transfer line onto the scrubber union using the 1/16" Teflon tube ferrule (Part #196220). 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.

CAUTION: The reactor is hot!

- Turn the solvent flow on using the DIP switch on the cell amplifier board.

- Set the solvent flow to the desired flow rate using the solvent flow adjustment on the cell amplifier board.

- Adjust the reactor temperature to the desired level, and allow the system to stabilize (approximately 5–10 minutes).

The 1/16" Teflon ferrule (Part #272443), 1/16" GRP/VSP tube ferrule (Part #216366), and 1/16" Teflon tube ferrule (Part #196220) 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" in this chapter). 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. Teflon 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 Figure 5.2). Common symptoms that indicate the need to replace the scrubber include: response to halogens, no response, low response, or blocked scrubber. Replace the scrubber as described in the following procedure.

Note: Do not install the scrubber until after the reacter tube has been conditioned for 30 minutes.

Note: The reactor should remain **hot** while this procedure is performed.

- Turn the solvent flow off.
- Turn the Model 5300 Detector Controller power off.
- Remove the scrubber union from the scrubber by loosening the scrubber tube nut between the scrubber and the union (see Figure 5.2). Inspect the Teflon tube ferrule (Part #196220) and replace, if necessary.
- Remove the scrubber by removing the knurled reactor nut at the top of the reactor. Examine the 1/16" Teflon ferrule (Part #272443) seated in the reactor nut and replace, if necessary.
- Install a new scrubber by inserting the knurled reactor nut and Teflon ferrule onto the scrubber and **finger-tightening** the reactor nut onto the reactor union. See Figure 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.

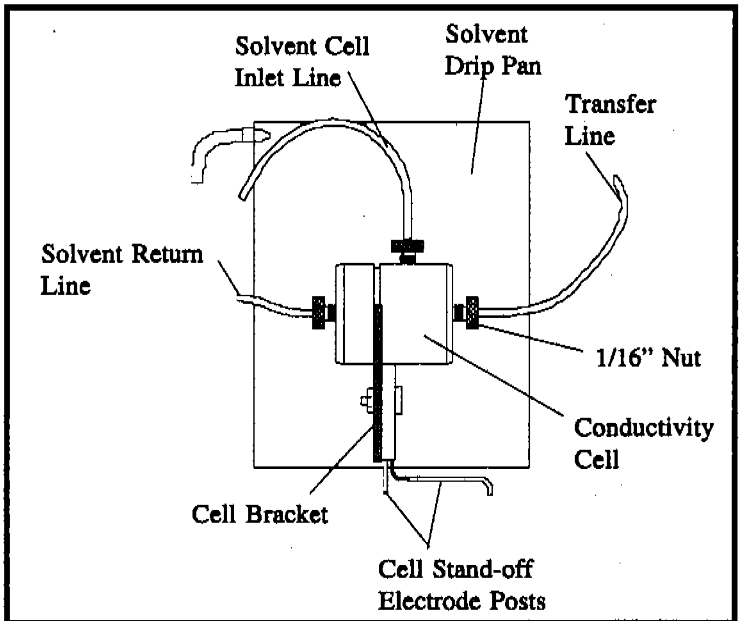

Figure 5.4. Conductivity Cell Assembly

Conductivity Cell Removal and Cleaning

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

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

- Turn the solvent flow off.
- Turn the Model 5300 Detector Controller power off.
- Remove the cell enclosure cover.
- Remove the three solvent lines from the conductivity cell by removing the three nuts. (see Figure 5.4).
- Remove the conductivity cell by carefully pulling it away from the two electrode posts.

Note: Do not take the conductivity 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 65°C.

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

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

Note: A conductivity cell that is not completely dry will cause a high baseline.

- Reinstall the conductivity cell by carefully inserting the two electrode posts into the conductivity cell.

- Connect the three solvent lines to the conductivity cell (see Figure 5.4). Do not overtighten the 1/16" nuts—**finger-tight** will suffice.

- Check the gas flow rates (see Chapter 4, "Setting the Gas Flows").

- Adjust the solvent flow to the desired flow rate and check for solvent leaks at the conductivity cell. If leaks occur, check the thumb nuts for proper tightness, but do not overtighten. If a leak occurs at a properly tightened nut, replace the Teflon ferrule (Part #272446) seated in the nut.

- Replace the cell enclosure cover.

Replacing the Reactor Base Adapter

If the reactor base adapter (Part #223743) becomes chipped, cracked, and/or deformed, it must be replaced according to the following procedure:

***CAUTION:** The nut is hot!*

- Turn the solvent pump and the Model 5300 Detector Controller power off.

- Remove the reactor (see "Replacing Reaction Tube" in this chapter).

- For tandem PID/ELCD, remove the reactor base adapter from the PID outlet using either the 1/4" nut driver supplied in the start-up kit or a 1/4" open-ended wrench. Replace with a new reactor base adapter.

- For stand-alone ELCD, unscrew the reactor base adapter and pull upward.

- Install a new reactor base adapter onto the reactor base.

- Reinstall the reactor with a new reaction tube (see "Replacing Reaction Tube" in this chapter).

- Turn the Model 5300 Detector Controller power on.

Notes

Chapter 6
Troubleshooting

The following is a list of the most common troubles that can occur when using the Model 5320 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, 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	No electrolyte flow	Repeat "Set the 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.
	Electronic device not connected properly	Review "Installing the Model 5300 Detector Controller" in Chapter 3. Correct any connection problems.
Low or Tailing Response	Carrier inlet line to cell contaminated	Refer to "Transfer Line Rinsing" in Chapter 5.
	Reaction tube fouled	Repeat "Replacing Reaction Tube" in Chapter 5.
	Leak in GC injector system	Check injection septum for leak. Inspect and tighten other components as needed.
	Cell contamination	Repeat "Cell Removal" in Chapter 5.
	Incorrect reactor temperature	Reset to specified temperature.
	Column degraded	Replace column. Refer to "Install Column" in Chapter 3.
	Reactor failed	Replace the reactor core.
High Baseline	Contaminated electrolyte	Repeat "Refilling Solvent," and "Replacing Resin Cartridge" in Chapter 5.

SYMPTOM	PROBABLE CAUSE	CORRECTIVE ACTION
	Conductivity cell wet or damaged	Dry conductivity cell in oven at 100°C for one hour, then retest.
	Column bleed	Cool column while monitoring baseline.
Spikes in Baseline	Reaction tube not conditioned	Allow reaction tube to condition at 950°C for ten 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.
	Overtightened solvent lines	Check solvent lines.
Noise on Peaks	Pulsating pump flow	Check pump and solvent flow rate. Replace pump.
	Dirty cell	Clean cell. See "Conductivity Cell Removal and Cleaning" in Chapter 5.
	Defective cell	Replace cell.
	Low solvent flow	Increase solvent flow.

Chapter 7
Replacement Parts

Throughout this manual, the various components of the Model 5320 ELCD 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
Board - PCA - 5320 Cell Amplifier	274266	ea	
Cable 5320 to HP 5890	246371	ea	
Cable Cell Interface	247007	ea	
Cable Reactor Power	245506	ea	
Cable Signal out to Spades	246405	ea	
Cable Signal out to HP Integrator	246397	ea	
Cartridge - Resin Hx Disposable 5320	213579	ea	*
Cell Assy - 5320 HP 5890	234237	ea	
Ferrule - 1/16" Tube GRP/VSP	196105	10/pk	*
Ferrule - 1/16" Tube GRP/VSP	196113	10/pk	*
Ferrule - 1/16" Tube GRP/VSP	208330	10/pk	*
Ferrule - 1/16" Tube Brass	223776	6/pk	*
Ferrule - 1/16" Tube GRP/VSP	216366	10/pk	*
Ferrule - 1/16" Tube Teflon	272443	5/pk	*
Kit - Halogen Mode 5320	219402	ea	*
Manifold Plate	197772	ea	
Manual - Model 5320 ELCD	274966	ea	
Nut - 1/16" Male Column	223057	ea	
Nut - 1/16" Stainless Steel (Reactor Top)	227462	ea	
Nut - 1/16" Stainless Steel Knurled	226357	ea	
Nutdriver - 1/4 Hollow	223917	ea	
O-Ring Manifold	185116	12/pk	*
Reactor Assembly - 5320	227496	ea	
Reactor Base Adapter	223743	ea	
Reactor - Core 5320	234187	ea	
Reactor Union	234021	ea	
Reactor Top Fitting	227462	ea	
Screw - Allen Set 6-32, 1/8"	131961	ea	*
Scrubber Union	204735	ea	
Seal - Reactor Base	223933	ea	
Solvent Pump Assembly - 5320	216267	ea	
Solvent Reservoir	216507	ea	
Tube - Alumina Reaction	217208	3/pk	*
Tube - Ni Reaction	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	*

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