

OPERATOR'S MANUAL

FOR

FTIR Interface

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Agilent Technologies

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Varian, Inc. offices

Varian, Inc. has offices in most countries. The major offices for chromatography products are listed below:

Varian, Inc. (Manufacturing site)
Essex Road
Church Stretton
Shropshire SY6 6AX, UK
International telephone: +44 1694 723581
International fax: + 44 1694 722171

Varian, Inc.
Scientific Instruments
2700 Mitchell Dr
Walnut Creek, CA 94598
Telephone: 1 800 926 3000
International telephone: +1.925.939.2400
International fax: +1.925.945.2360

Varian B.V.
Postbus 8005
4330 EA Middelburg, The Netherlands
International telephone: +31.118.67.1000
International fax: +31.118.62.3193

Varian Australia Pty. Ltd
679 Springvale Rd, Mulgrave,
Victoria 3170, Australia
International telephone: +61.3.9560.7133
International fax: +61.3.9560.7950

Varian Industria e Comercio Ltda.
Avenida Dr. Cardoso de Mello 1644
Sao Paulo 04548-005, Brazil
International telephone: +55.11.3238.0400
International fax: +55.11.3238.0410

www.varianinc.com

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eumktg@varianinc.com

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(Extract from General Conditions of Sale)

Subject as hereinafter stated, if any goods supplied are proved to the reasonable satisfaction of the Seller to be defective in material or workmanship within a period of 12 months from the date of despatch and the Buyer notifies such defect to the Seller in writing within fourteen days of it becoming apparent the Seller shall repair or replace at its option the goods or any part thereof free of charge and any repaired (or replacement) goods will be guaranteed on these terms for the unexpired portion of the 12 month period **PROVIDED THAT** the Seller shall be under no liability in respect of any defect that has arisen because:-

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- b. where the goods have not been used, maintained, stored or protected in the proper manner; or
- c. the goods have been altered in any way whatsoever or have been subject to unauthorised repair; or
- d. the goods have been improperly installed or connected (unless the Seller carried out such installation and connection); or
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- f. the Buyer is in breach of any other contract made with the Seller such as the Company's General Conditions of Sale.

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Any statement made about the Seller's goods by the Seller or its servants or agents whether orally or in writing is intended for guidance only and the Buyer should not place any reliance thereon without specific enquiry and without ensuring that any matter of concern to him is specifically mentioned in the contract.

The Buyer is solely responsible for the suitability of the site for the installation of the goods, for obtaining all and any necessary consents and approvals under planning and building regulations and by-laws and for the preparation of the site, the constitution of foundations and the provision of services so that the site is suitable to receive the goods.

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HEALTH AND SAFETY

FTIR Interface

The use of this instrument does not entail any hazard if used in accordance with the instructions stated within the Operator's manual. If the instrument is used in a manner not specified by the manufacturer, the protection provided may be impaired. Users should observe the following safety precautions:

- . Ensure that the instructions within the Operator's manual are understood and carried out in the operation of the instrument. All persons utilizing the instrument should have adequate training in its proper set-up, operation, and particularly its safety features.
- . Voltages above 110V AC are present within the instrument, and access covers should not be removed by anyone other than properly trained personnel. No attempt should be made to service the instrument without authorisation from Varian's service department and contravention of this may result in personal hazard or damage to the instrument and will invalidate the manufacturer's warranty.
- . We stress the importance of standard safe practice (such as COSHH regulations) for dealing with electronic laboratory equipment, solvents, etc., in preventing accidents, fires, or potentially hazardous conditions.

If in any doubt about the use of the instrument contact your local Varian office or local distributor.

In the UK and Europe

Varian, Inc.

Technical Assistance

Tel +44 1694 723581
Fax +44 1694 722171

Service

Tel +44 1694 724333
Fax +44 1694 723994

United States

Varian, Inc.

Technical Assistance & Service

Tel, toll free 800 767 3963
Tel (413) 253 9554
Fax (413) 253 2476

Australia

Varian Australia Pty. Ltd

Sales & Service

Tel +61 3 9566 1134
Fax +61 3 9566 1471

In the Benelux countries

Varian B.V.

Technical Assistance & Service

Tel +31 118 671500
Fax +31 118 671502

In Germany

Varian Deutschland GmbH

Technical Assistance & Service

Tel +49 6151 7030
Fax +49 6151 703237

In France

Varian SA

Technical Assistance & Service

Tel +33 1 6986 3838
Fax +33 1 6928 2308

SAFETY

Signs and Pictograms Used in this Manual



WARNING

WARNING:

The “WARNING sign” denotes a hazard. It calls attention to a procedure or practice which, if not correctly done or adhered to, could result in severe injury or damage or destruction of the instrument. Please do not proceed beyond a WARNING sign until the indicated conditions are fully understood and met.



ATTENTION

ATTENTION:

The “ATTENTION sign” denotes relevant information. Read this information first before proceeding, it will be helpful or necessary to complete the task.



NOTE

NOTE:

The “NOTE sign” denotes additional information.

It provides the user with advice and suggestions to facilitate the operation of the instrument.

Safety Practices

The following general safety precautions must be observed during all phases of operation, service, and repair of this equipment. Failure to comply with these precautions or with specific warnings elsewhere in this manual violates safety standards of design, manufacture, and intended use of the equipment. Varian assumes no liability for the customer's failure to comply with these requirements.

THERE ARE NO USER-SERVICABLE PARTS INSIDE THIS EQUIPMENT. IN THE EVENT OF FAILURE CONTACT VARIAN'S SERVICE DEPT.



WARNING

Electrical Hazards

Removal of instrument panels may expose potentially dangerous voltages. Disconnect the instrument from all power sources before removing protective panels.

Replace defective fuses **only** with size and rating stipulated on the rear panel next to the fuse holder, and in the manual.

Replace faulty or frayed power cords.

Check the actual line voltage to confirm its value before connecting this instrument to it.



WARNING

Cleaning

The individual or group responsible for the use and maintenance of this equipment must ensure that appropriate decontamination is carried out if hazardous material is spilt on or inside the equipment.

Before using any cleaning or decontamination method except those recommended by Varian, operators should check with Varian that the proposed method will not damage the equipment or cause a hazard as a result of a reaction with parts of the equipment or with material contained in it.

See Chapter 5 for detailed recommended procedures.



NOTE

General Precautions

Regularly test the overtemperature protection system.

Perform periodic leak checks on all wetted components, tubing and joints.

Do not allow flammable and/or toxic solvents to accumulate.

Follow recommended procedures and protocols for evacuation and disposal of flammable and/or toxic solvents. Never dispose of such products through municipal waste systems



DECLARATION OF CONFORMITY

We, Varian, Inc.
Essex Road
Church Stretton
Shropshire SY6 6AX
U.K.

certify that the following products:

FTIR Interface

Part Numbers PL0820-2200, PL0820-2220, PL0820-2280, PL0820-2140, PL0820-2100, PL0820-2120, PL0820-2180

conform with the requirements of EC Directives 98/37, 73/23, 93/68, 89/336 & 92/31 by complying with the following Harmonised European Standards:

Safety:	BS EN61010-1:1998	Class I Installation category II Pollution degree 2 REINFORCED INSULATION
	BS EN61010-2-010:1995	

EMC:	BS EN61326:1998	Performance Criterion: A Conducted & Radiated Emissions: Class B
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Dr. S.O'Donohue
Head of Instrumentation, Church Stretton
8th June 2010

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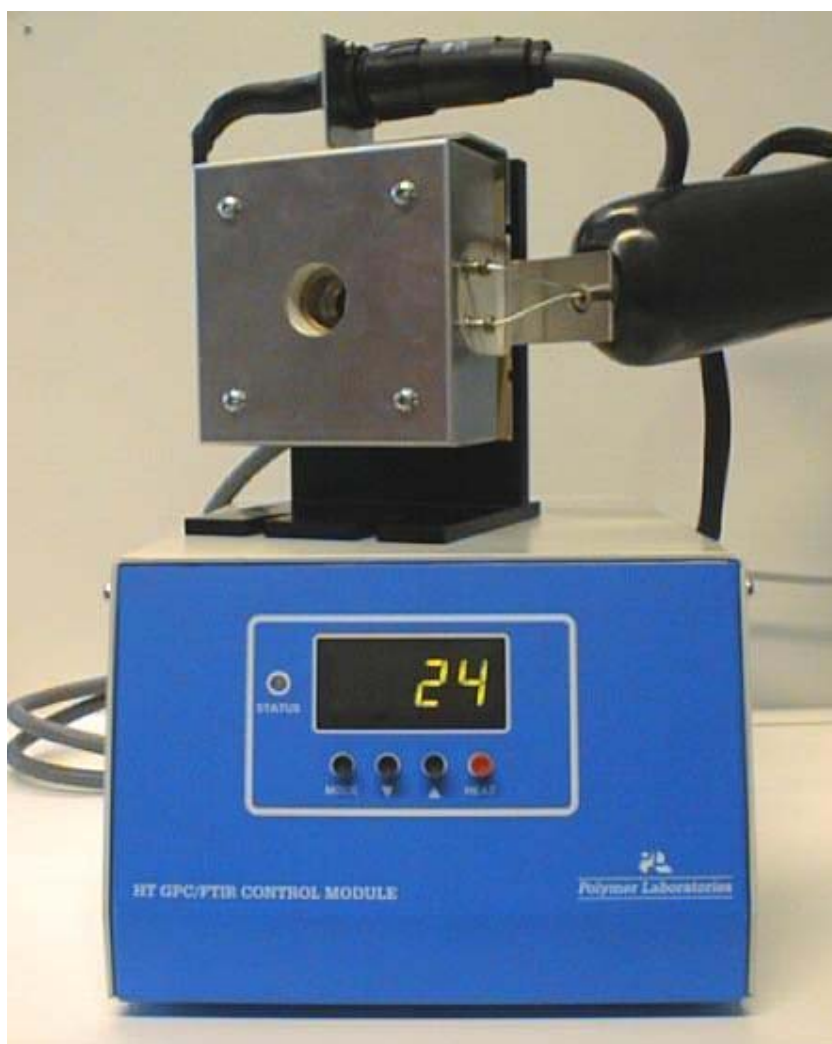
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1 General Information

1.1 Overview

The FTIR Interface comprises a heated flow cell, fitting kit, heated transfer line and control module. The interface is designed for use with a modern FTIR spectrometer, which are available from Perkin-Elmer or Nicolet



1.2 Specifications

Power Requirements:	System:	95/125V or 210/250V AC 50/60Hz Maximum current 5A
Environmental Conditions	Operating:	Indoors, 10 °C – 30 °C, 80% R.H. max.
Temperature Range	Cell & Transfer line	Ambient to 175 °C
Temperature Accuracy	Cell Transfer line	±0.5 °C ±0.5 °C
Temperature Control	Cell & Transfer Line	PID Control with RTD temperature sensor
Temperature Stability	Cell Transfer Line	±0.1 °C ±0.3 °C
Cell Volume		70 µL
Path length		1 mm
Windows		Calcium fluoride (16 mm Dia x 4 mm thick)
Maximum back pressure	Cell	5MPa
Body Construction		316 stainless steel with Valco 1/16" low dead volume fittings
Size	Cell assembly Controller	170 x 150 x 120 mm (HxWxD) 136 x 200 x 250 mm (HxWxD)
Weight	Cell assembly Controller	2.3 kg (5.1 lbs) 2.8 kg (6.2 lbs)
Safety	Cell Transfer Line	Secondary RTD sensor with independent over-temperature cut-out preset to 185 °C Thermal fuse, rated at 240 °C
Accessory Ratings:	Cell Transfer Line	230V AC 50/60Hz 600W 230V AC 50/60Hz 230W MAX

1.3 Unpacking and Installation

Care has been taken to ensure that this equipment should be received in proper condition. The packing and protection are designed for normal hazards of road, rail or air transit. Any damage to the container or instrument should be reported immediately to your local distributor or to Varian. It is recommended that the shipping container be kept, if possible, for re-shipment or return to a service centre.

Examine the shipping box for visible signs of exterior damage. Unpack the instrument and examine for transit damage. Check that all items on the packing list are included.

Notify your local distributor or Varian of any damage or missing items.

1.3.1 Packing list

- . Flow cell (pre-assembled and mounted on bracket)
- . Heated transfer line
- . Temperature control module
- . Mains Lead

1.4 Location

The FTIR flow cell is designed to be mounted inside the cell compartment of a suitable FTIR spectrometer. The control cable and heated transfer line should exit the cell compartment from the top or the side to suit the set-up. Please provide adequate space around the temperature control module to allow ventilation of the electronics.

1.5 Utility Requirements

1.5.1 Power Connections

- ◆ The unit requires a single phase AC supply, either 110V or 230V nominal, 50/60Hz, 5A.
- ◆ The mains supply should be fitted with an RCCB.
- ◆ **Ensure the power switch and appliance coupler remain accessible at all times.**
- ◆ Before plugging in the power cable, ensure the voltage selector on the IEC mains matches your local power supply.
- ◆ Use only a supply with **PROTECTIVE GROUNDING**.
- ◆ The unit is double fused (line and neutral) and the correct fuses should be installed in the IEC inlet:

Two 5A F 250V HBC fuses



RISK OF FIRE, REPLACE FUSES AS MARKED!

1.6 Precautions

1.6.1 Temperature

The FTIR cell and heated transfer line are designed to be controlled at temperatures up to 175 °C and therefore caution should be exercised when handling these components.



BEWARE HOT SURFACES !

1.6.2 Cell and Transfer Line Fuses

Individual fuses protect the cell and heated transfer line. These fuses are of ultra-fast type to prevent damage to the semiconductor power control electronics. Ensure that replacement fuses are of FF type:

Cell fuse: 3.15A 250V FF HBC

Transfer Line Fuse: 1A 250V FF HBC

1.6.3 Drying-out Period

After assembling the equipment, and before its first use, the cell should be allowed to run at 150 °C for approximately one hour, **without adding solvent**, to allow any internal moisture to be dried out from the cartridge heaters. The equipment cannot be assumed to meet all relevant safety standards until this process is completed. If the liquid connections are fitted with nylon blanking plugs, remove these before heating. One of the liquid connections should be loosened to allow any residual solvent to vent (loosen the top fitting on side connection versions).

2 System Overview

The FTIR Interface is illustrated in Figure 2.1. It comprises:

- heated flow cell
- heated transfer line
- temperature control module

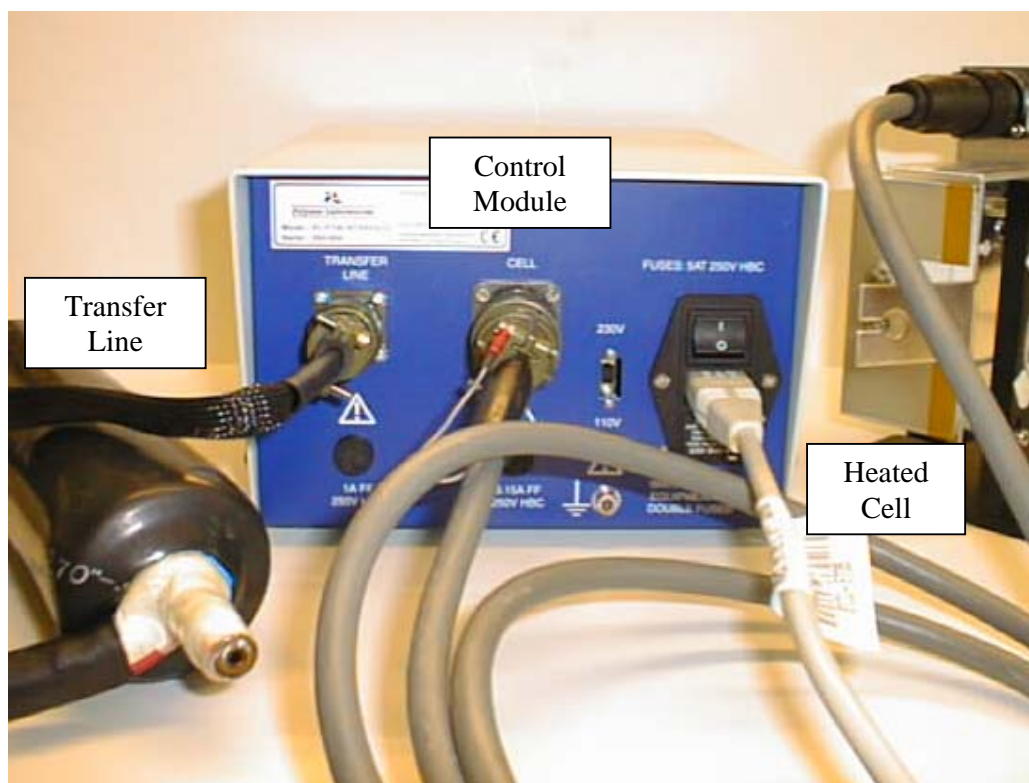


Figure 2.1 System Overview

3 Set-up

Before fitting the cell into the spectrometer, connect the heated transfer line to the cell assembly and mount the cell on the bracket provided

3.1 Assembling the cell

The cell is usually assembled during manufacture, pressure tested to 5MPa, and therefore arrives ready to install. Figure 3.1 below shows an exploded view of the assembly should it be necessary to disassemble the unit.

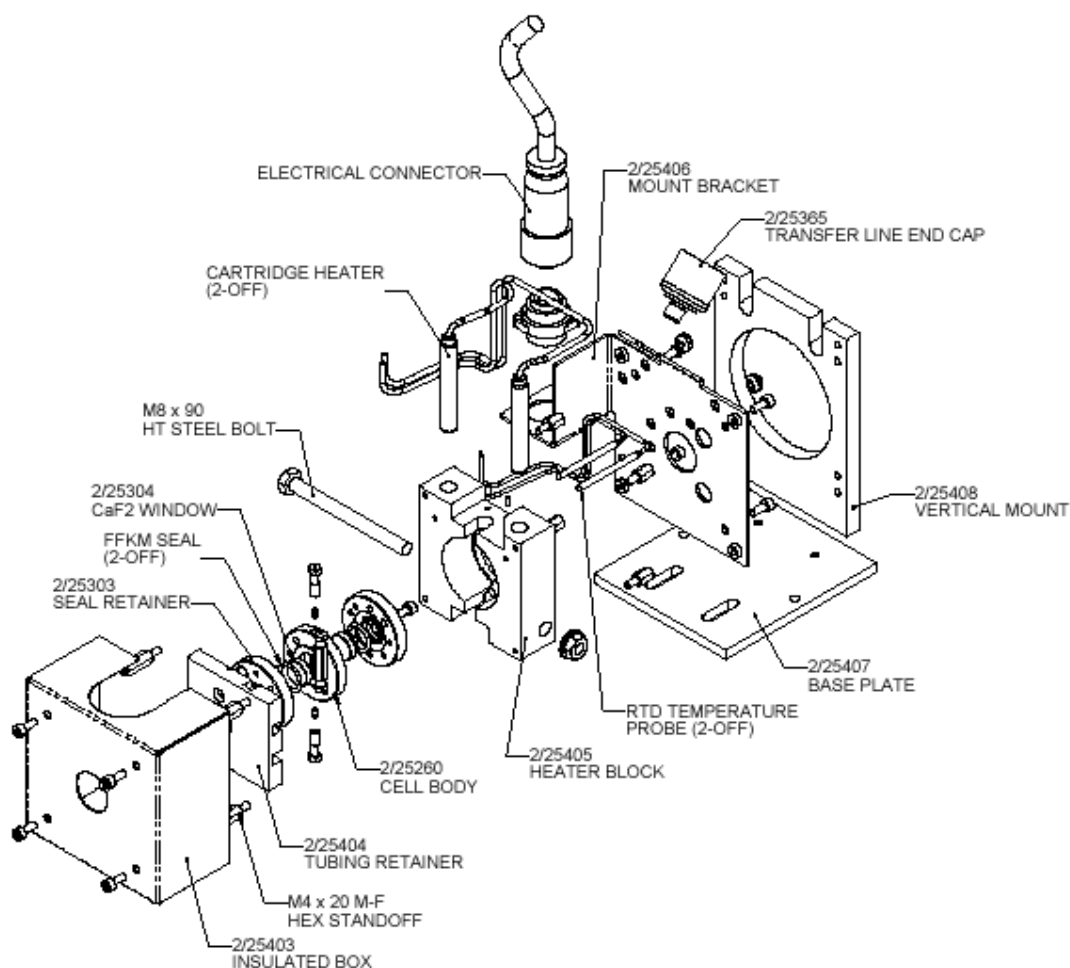


Figure 3.1 Exploded Diagram of the Flow Cell

The cell can be assembled at the factory in one of two alternative configurations;

- A. The liquid connections at the side
- B. The liquid connections at the top.

The cell body is manufactured so the additional connecting tubing in these alternative configuration is inlaid into the heated block. Figures 3.2 and 3.3 show configurations A and B respectively.

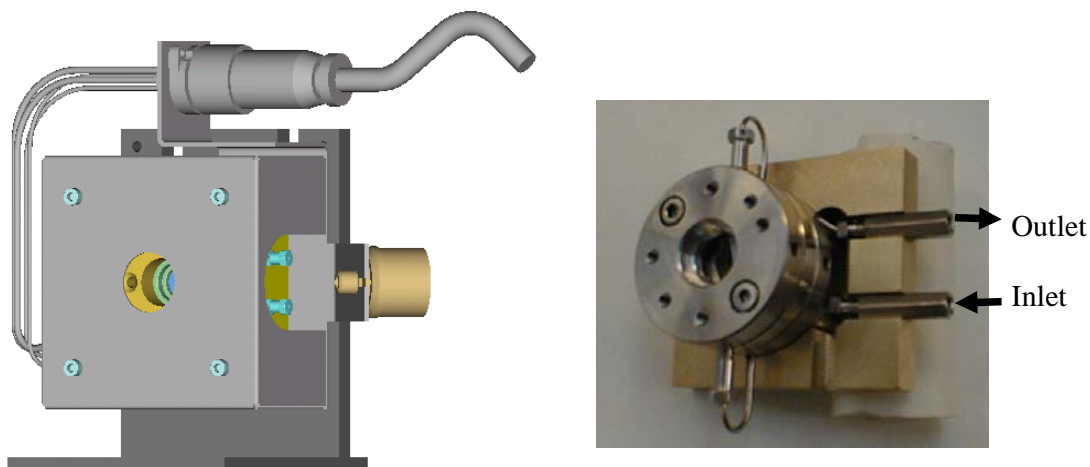


Figure 3.2 Liquid Connections at the Side

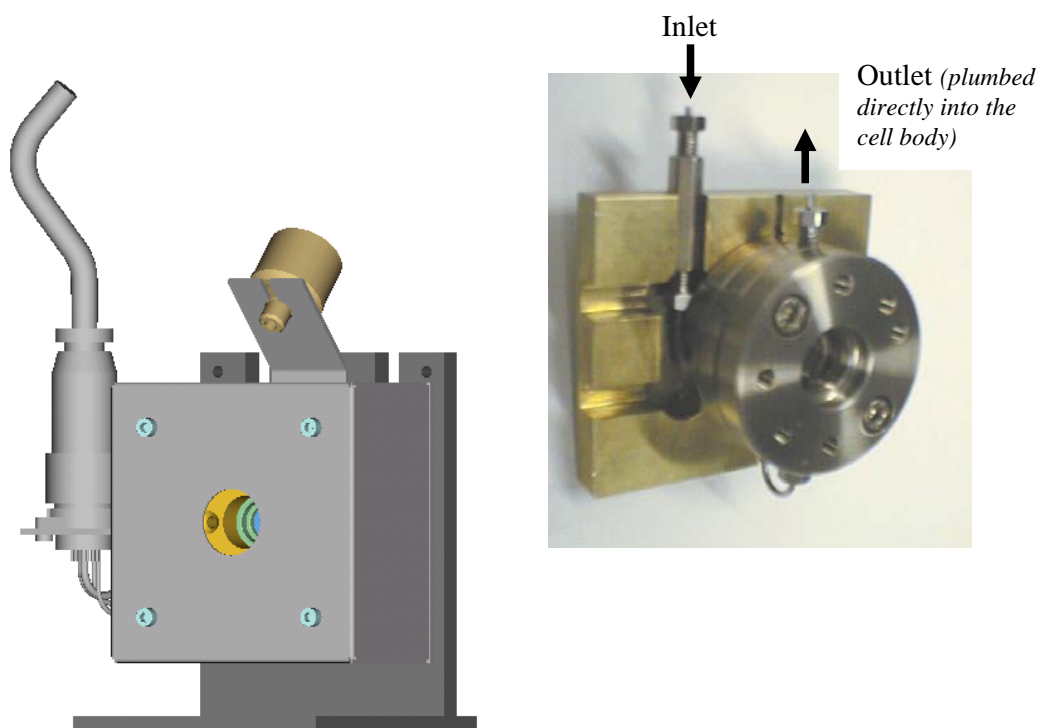


Figure 3.3 Liquid Connections at the Top

3.2 Fitting the Heated Transfer Line

The heated transfer line is lined with a length of copper tubing making fitting and removing the stainless steel capillary tubes very easy, even after the line has been bent. The heated transfer line will accommodate 3 x 1/16 in. OD capillary tubes, which would be typically;

- 2 x 0.01 in. ID tubing, one for the inlet and a spare
- 1 x 0.02 in. ID tubing for the outlet

1. Carefully slide the desired number and size of capillary tubes into the heated transfer lines. Ensure that the capillaries are long enough so that connections can be made to the required equipment at both ends of the line.
2. Remove the front cover from the flow cell by removing the four screws located at each corner of the cover (Figure 3.4)

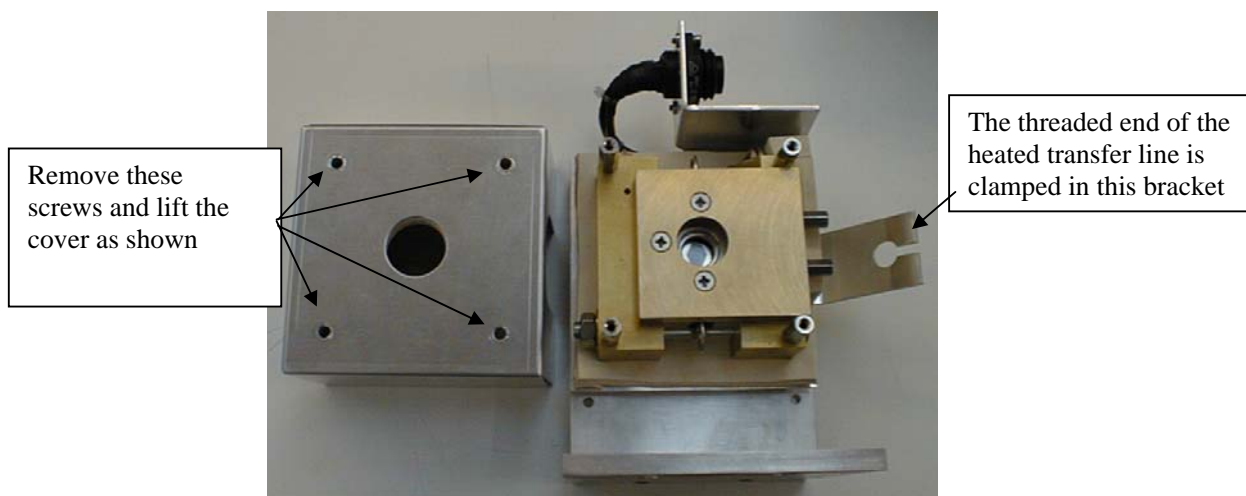


Figure 3.4 Flow cell with the cover removed

3. Connect the capillaries to the cell. On a side connection the inlet should be plumbed to the lower fitting and the outlet to the upper connection. When the line is configured to the top of the cell the inlet should be plumbed to the barrel connector provided and the outlet direct to the cell.



Use long bushings when connecting directly to the cell and use Valco fittings for both the cell and the connectors

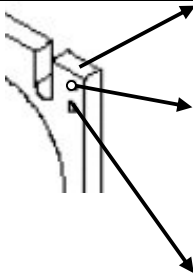
4. Slacken the nut on the end of the heated transfer line and slip the tubes into the slot in the cell bracket. Take up the slack in the tubes by pulling them through the line and insert the threaded end of the heated transfer line into the bracket and tighten the nut (Figure 3.4).
5. Insulate the exposed parts of the SS capillaries before refitting the front cover.
6. Connect the cell control cable to the connector on the cell.

3.3 Fitting the Cell assembly into the Spectrometer

1. Position the cell in the spectrometer so that the fixing screws locate with the mounting holes in the spectrometer base plate.
2. Before tightening the screws down, centrally align the cell into the optical path.
3. Final alignment of the cell is carried out in conjunction with the FTIR control software so that the spectrum energy is maximized.

3.4 Beam Height

The cell can be positioned in one of three positions so that the centre of the cell can be accurately aligned with the centre of the beam within the spectrometer. The height of the cell centre for each of the three positions are given in the table with the recommended position for most popular spectrometers.

	Mounting Hole Positions	Height of cell centre (mm)	Compatible FTIR Spectrometer
	Top	88	Nicolet Protégé Nicolet Avatar
	Middle	83 80	PE Spectrum One Bruker Tensor 27 (when mounted on Bruker plate)
	Bottom	77	PE System 2000

3.5 Electrical connections



ENSURE CONTROLLER IS ISOLATED FROM MAINS POWER BEFORE CONNECTING OR DISCONNECTING TRANSFER LINE OR HEATED CELL.

Figure 3.5 below shows the rear panel of the Temperature control module. All the connectors are clearly identified and the following cables are to be connected to the sockets indicated.

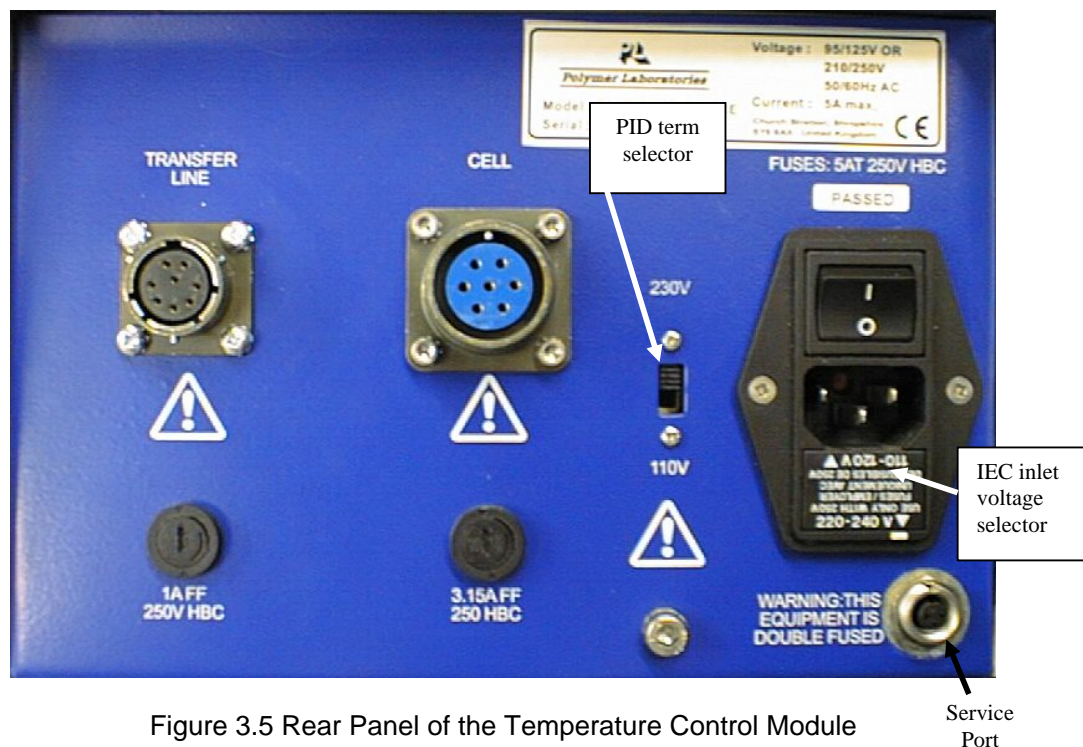


Figure 3.5 Rear Panel of the Temperature Control Module

Cable	Socket on Rear Panel	Function
Control cable from Cell	CELL	Carries the power to the cell heaters and the temperature feedback from the control and safety RTDs
Circular plug from HTL	TRANSFER	Carries the power to the transfer line heater and the temperature feedback from the control RTDs
Mains Lead	IEC Inlet	Mains power for control module and heaters
No connection	Service Port	For service use only



**USE ONLY HEATED CELLS AND TRANSFER LINES SUPPLIED BY
VARIAN FOR USE WITH THIS CONTROLLER.**

3.6 Line Voltage Selection

The standard heated cells and transfer lines supplied by Varian will operate from both 110V and 230V supplies. Two operations are required to configure the Temperature Control Module to your local line voltage:

1. Ensure that the voltage shown at the bottom of the IEC inlet voltage selector matches your local line voltage. If it does not match, prise the voltage selector out of the inlet body and replace it in reverse orientation.
2. Ensure that the PID term selector switch is positioned to match your local line voltage.

If you have been supplied with a custom (long) transfer line then the unit will only operate from a 230V supply.

4 System Operation

4.1 Temperature Control Module

Figure 4.1 shows the front of the temperature control module. It features a temperature display which reports the temperature of the cell, a heat on and off button and a mode button to change the display to the temperature set point and enable the temperature adjustment buttons.



Figure 4.1 Temperature Control Module

4.1.1 Switching the temperature control module on.

The power switch for the temperature control module is located at the rear of the box, positioned above the mains inlet cable. Switching the rocker switch to position I should illuminate the temperature display and the temperature of the cell will be displayed.

4.1.2 Setting the control temperature

The cell and the heated transfer line are controlled at the same temperature. Normally the cell temperature is displayed on the control module.

To set the control temperature press the **Mode** button once to display the set temperature and adjust by using the **Up** and **Down** buttons.

If the **Mode** button is pressed again before this period has elapsed then the display will show the transfer line temperature. The status LED will be continuously Red to distinguish this.

The display will revert back to the cell temperature 5-6 sec after the last key-press.

4.1.3 Switching On the heaters

The heaters in the cell and heated transfer line are turned On by pressing the **Heat** button. The LED to the left of the display indicates the heaters are operative by flashing (amber) if the cell or line is heating or cooling and on continuously (green) if the cell and line are at the set temperature. Pressing the **Heat** button again switches the heaters Off.

Status LED:

- Flashing Amber while heating or cooling to new set point
- Green continuously while at set point
- Red continuously while showing transfer line temperature
- Off when heaters are off

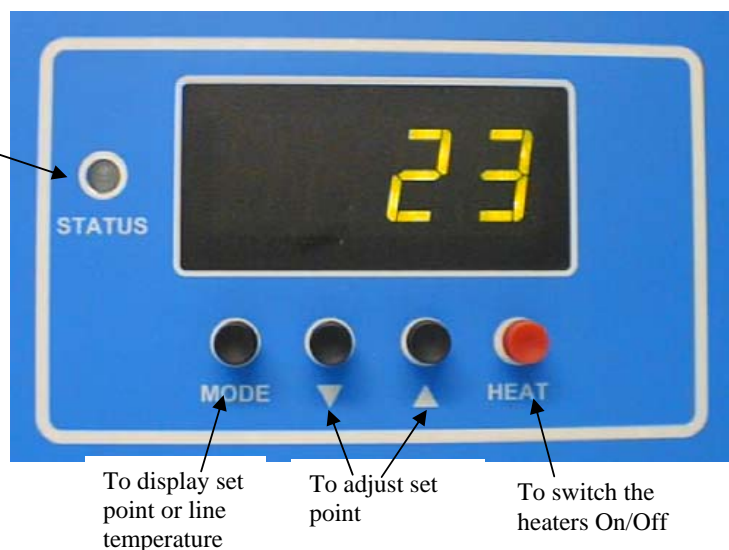


Figure 4.2 Temperature Display and Control Buttons

4.1.4 Error Conditions

Should the recorded temperature overshoot the set point, or if one of the temperature sensors are disconnected, an audible alarm will sound accompanied by a flashing temperature display to indicate an error condition. To reset, power the module off and then on again.

4.2 Safety Temperature Cut-Out

The FTIR interface is protected against thermal runaway by employing an independent secondary temperature system which will switch off the heaters should the cell temperature reach 185 °C. The heated transfer line is protected by a thermal fuse which will open and cut the power to the heater if the line reaches 240 °C.

5 **Cleaning and Maintenance**



WARNING

SWITCH INSTRUMENT OFF AND DISCONNECT POWER CORD BEFORE UNDERTAKING ANY CLEANING OR MAINTENANCE PROCEDURES. ALLOW ANY HOT PARTS TO COOL BEFORE PROCEEDING.

5.1 Cleaning

The exterior of the instrument should be cleaned by wiping down with a soft cloth moistened with dilute detergent solution, followed by wiping down with a cloth moistened with deionized water.

- Ensure that no moisture enters the instrument.
- Do not attempt to clean any electrical connectors. Ingress of moisture could cause a safety hazard and accessible pins may become bent.
- Allow exterior of instrument to dry off completely before reconnecting power.

5.2 Decontamination

THIS PROCEDURE TO BE CARRIED OUT BY TRAINED SERVICE PERSONNEL ONLY

The operator should wear appropriate personal protective equipment for this operation (gloves, safety glasses, lab coat and respirator if level of hazard has been risk-assessed to be sufficiently high).

In the case of a contaminated cell assembly, this should be taken apart and cleaned (in a fume cupboard in necessary) following the disassembly instructions in this manual. If the control interface or transfer line are contaminated, seek advice from Varian's Service Dept.

Remove instrument covers and panels as necessary to ascertain the extent of contamination. Excess quantities of solvent spilt on or inside the instrument should be mopped up using absorbent cloths, followed by repeated wiping down with soft cloths moistened with acetone until the last traces of the hazardous liquid have been removed.

Identify and, if possible, correct the source of the leak. Inspect cabling, parts and surfaces to determine whether any damage has occurred. If in any doubt, contact Varian's Service Dept for assistance before reassembling & reconnecting the instrument.

Allow the interior and exterior of the instrument to dry out completely before replacing panels and covers and reconnecting power.

Dispose of contaminated waste appropriately.

5.3 Testing the Cell Safety Temperature Cut-out

The heated cell is fitted with an independent overtemperature protection system to eliminate potential hazards due to failure of the temperature controller. The trip point is fixed at 185 °C. Correct operation should be tested regularly.

To test the protection system:

- **The cell and transfer line must first be filled with deionized water.** It may be necessary to transfer solvents through Acetone. If so, cool the cell and transfer line to 40 °C beforehand.
- Ensure the fluid outlet tube at the end of the transfer line furthest from the cell is open to atmosphere. It is advisable to route the open end into a small vessel to collect any drips from fluid expansion and vaporization. Check that this outlet is open by flushing water through the cell from the inlet end and observing liquid emerging into the vessel. See pictures below.



A PRESSURE HAZARD WILL EXIST IF THE END OF THE OUTLET TUBE IS CLOSED OFF DURING THE REMAINDER OF THIS PROCEDURE.

- Set the temperature to 186 °C or 187 °C (it may be necessary to “wrap-around” to 30 °C and go back down). Press the **Heat** button and wait for the temperature to rise.
- As the cell temperature approaches and passes 100 °C a few drops of water will emerge from the open end of the outlet tube.
- When the cut-out is activated at 185 °C, an audible alarm will sound and the display will flash. Do not switch off yet.
- If the alarm does not sound: first check that the above procedure has been followed correctly. **If the procedure has been carried out correctly but no alarm sounds, disconnect the power cord from the instrument and contact Varian’s Service Dept.**
- The power to the cell heaters and also the transfer line is now interrupted, so it is necessary to wait until the displayed temperature of both falls in order to verify correct operation of the protection system (press the **Mode** button to check).
- **If the displayed cell and transfer line temperatures show no decrease after 5 minutes while the alarm is sounding then switch the instrument off, disconnect the power cord and call Varian’s Service Dept.**
- Once cooling has been verified, switch the control interface off at the mains power switch to silence and reset the alarm.
- Allow the cell and transfer line to cool to before transferring back to the desired solvent (transfer via Acetone may be necessary). Reconnecting the inlet and outlet tubing to the chromatography system.
- This concludes the temperature cut-out test.



5.4 Removing the Flow Cell

1. Turn off the temperature control module and unplug the unit from the mains.
2. Remove the cell assembly from the spectrometer.
3. Disconnect the heated transfer line, and disconnect the electrical connections from the cell.
4. Remove the front cover by removing the four screws at each corner (Figure 5.1).

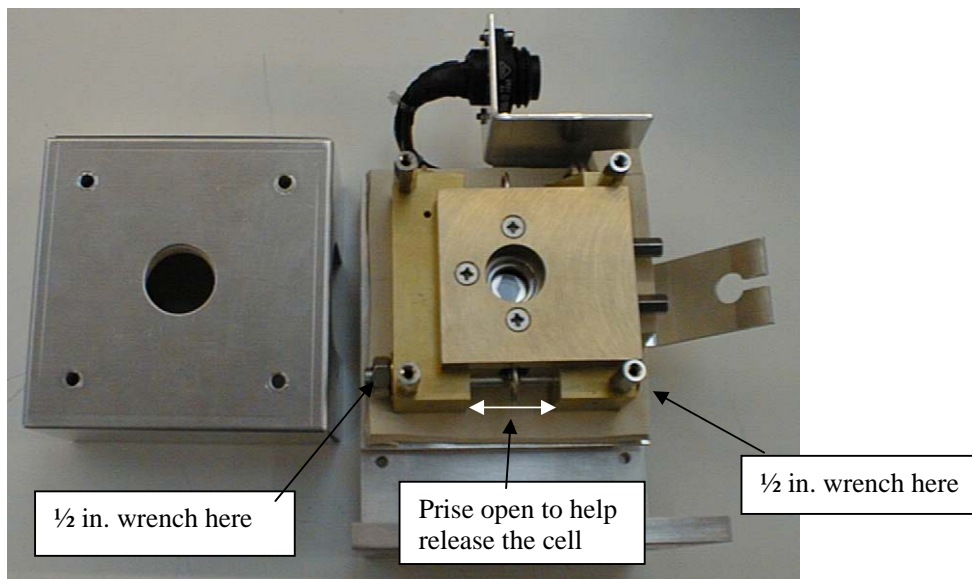


Figure 5.1

5. Using two 1/2 in. wrenches loosen the bolt below the cell. The brass square section to which the cell is bolted can now be teased out of the heated block. Prising open the brass heated block by the clamp screw helps release the cell.

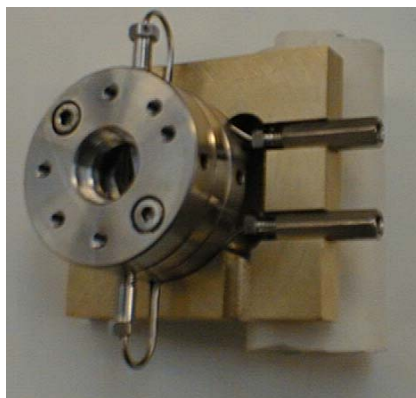


Figure 5.2

6. To remove the cell from the brass plate remove the three screws that are seen on the brass plate side of the assembly. The barrel connectors can be prized out of the slots, enabling the cell complete with tubes to be removed.

5.5 Disassembly of the Cell

1. To disassemble the cell to exchange the cell windows or seals, remove the 4 socket headed screws (2 from one side and 2 from the other) from the cell body.
2. The cell will now come apart as shown in Figure 5.3 below.

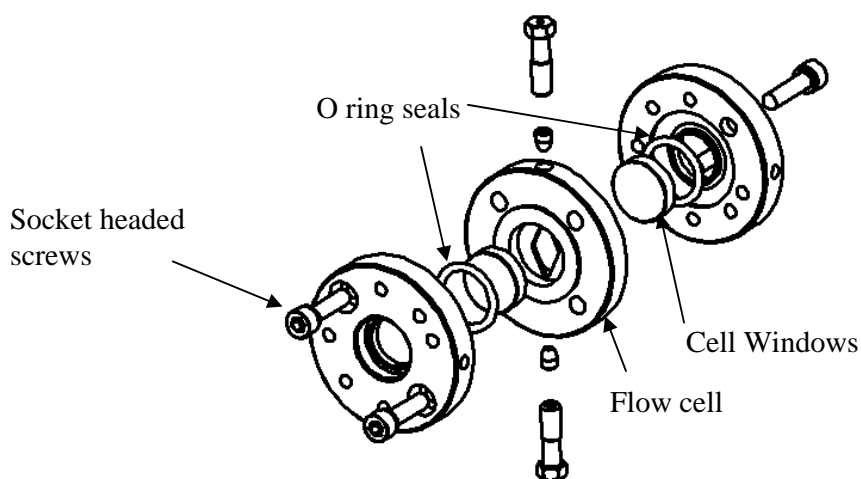


Figure 5.3 Exploded diagram of the cell body

3. If the cell windows are difficult to remove, applying a small amount of pressure to the inside of the cell via the liquid lines will help force the windows out.

5.6 Reassembly of the flow cell

4. The flow cell is assembled in reverse of the disassembly procedure described above, but ensure the hole pattern required to fix the cell to the brass plate is in the correct orientation.
5. The 4 socket head screws bolting the cell body together can be fitted in any order, but tighten each a little at a time so the cell pulls together squarely. The screws can and should be torqued up very tightly, without fear of cracking the windows.

5.7 Inspection of Cables

Periodically inspect the connecting cables for signs of physical damage caused by abrasion, solvent spillage, impact etc. Replace cables if any damage is observed.

5.8 Information for Service Personnel

THERE ARE NO USER-SERVICABLE PARTS INSIDE THIS EQUIPMENT. IN THE EVENT OF FAILURE CONTACT VARIAN'S SERVICE DEPT.



WARNING

DISCONNECT POWER CORD BEFORE REMOVING COVER!

The following internal fuses are fitted:

F1	F 4A 250V TR5
F11	T 2A 250V (PSU)