Varian BV
Herculesweg 8
4330 EA Middelburg
The Netherlands

450-GC

User Manual

North/South America
2700 Mitchell Drive
Walnut Creek
94598 California, USA
Tel: ++(1)9259392400
Fax: ++(1)9259452360 or
++(1)9259452344

Europe
P.O. Box 8033
4330 EA Middelburg
The Netherlands
Tel: ++(31)118671000
Fax: ++(31)118623193

Australia/East Asia
679 Springvale Road
Mulgrave, Victoria 3171
Australia
Tel: ++(61)395607133
Fax: ++(61)395607950

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Read Before Operating

Important Safeguards

The following items are frequently not recognized or are overlooked by new users while learning to operate a Gas Chromatograph.
They are brought to your attention to safeguard against damage to your equipment.

1. Carrier gas must be flowing through the column before heating the column oven. Carefully evaluate columns that have been heated with no carrier gas flowing through and discard, if damaged. Ensure the Injector contains a septum and there is a positive flow of carrier gas before heating the column.

2. Become familiar with the location and identity of Injector and detector options prior to making settings at the GC keyboard and pneumatics panel. For example, determine the physical location of the Injector you want to use (front, middle, or rear) and identify the specific pneumatic controls associated with it (Electronic Flow Control). Similarly, determine the physical location of the detector you want to use and identify the specific pneumatic controls for this detector.

3. Heating an ECD to elevated temperatures without carrier or make-up gas flowing through it can damage the ECD cell. This can happen inadvertently especially when the ECD is not in use. For this reason, it is recommended that a low flow of carrier or make-up gas be maintained through an ECD at all times.

4. If your system has a TCD, gas must be flowing through both channels to protect the filaments. Turn power to filaments off when not in use.

Important Tips Regarding 450-GC Operation

- If a potentially hazardous Error is detected, such as a thermal runaway, the 450-GC shuts down the affected component and reports the Error on page 84. After correcting the Error, normal operation is restored by powering the 450-GC OFF then ON.

- Using cryogenic column oven cooling, on page 73 leave always the Injectror(s) / Detector(s) heating ON (enable) and at a temperature of at least 100 °C.

VARIAN ANALYTICAL INSTRUMENT WARRANTY

For Instrument Warranty in German, French, Italian or Spanish please refer to the 430-GC, 431-GC and 450-GC User Manual CD-rom (CP501414) which is included with the GC.

HARDWARE PRODUCTS

All analytical instruments sold by Varian are warranted to be free from defects in material and workmanship for the periods specified and in accordance with the terms on the face of Varian's quotation or as otherwise agreed upon in writing between Varian and the Customer. The warranty period begins on the date of **shipment** from Varian to the original Customer. However, where installation is paid for by the Customer or included in the purchase price, the warranty period begins upon completion of installation. If the Customer schedules installation to start later than 30 days after delivery or if such delay is caused through the Customer's inability to provide adequate facilities or utilities or through failure to comply with Varian's reasonable pre-installation instructions or through other omissions by Customer, then the warranty period starts on the 31st day from date of shipment. Moreover Varian will charge the Customer for labor and other expenses involved in making multiple or follow-up installation service calls.

SOFTWARE PRODUCTS

Where software is provided within the frame of a license agreement concluded between the Customer and Varian, any warranty shall be strictly in accordance with the terms of such agreement.
In the absence of a license agreement and unless an alternate warranty period is agreed upon in writing between Varian and the Customer, the warranty period is as specified on the face of Varian's quotation. Varian warrants such software products, if used with and properly installed on Varian hardware or other hardware as specified by Varian to perform as described in the accompanying Operator's Manual and to be substantially free of those defects which cause failure to execute respective programming instructions; however, Varian does not warrant uninterrupted or error-free operation.

REMEDIES

The sole and exclusive remedy under hardware warranty shall be **repair** of instrument malfunctions which in Varian's opinion are due or traceable to defects in original materials or workmanship or, at Varian's option, **replacement** of the respective defective parts, provided that Varian may as an alternative elect to **refund** an equitable portion of the purchase price of the instrument or accessory.
Repair or replacement under warranty does not extend the original warranty period.
Repair or replacement under warranty claims shall be made in Varian's sole discretion either by sending a Customer Support Representative to the site or by authorizing the Customer to return the defective accessory or instrument to Varian or to send it to a designated service facility. The Customer shall be responsible for loss or damage in transit and shall prepay shipping cost. Varian will return the accessory or instrument to the Customer prepaid and insured. Claims for loss or damage in transit shall be filed by the Customer. To correct software operation anomalies, Varian will issue software revisions where such revisions exist and where, in Varian's opinion, this is the most efficient remedy.

LIMITATION OF WARRANTY

This **warranty does not cover** software supplied by the Customer, equipment and software warranted by another manufacturer or replacement of expendable items and those of limited life, such as but not limited to: Filters, glassware, instrument status lamps, source lamps, septa, columns, fuses, chart paper and ink, nebulizers, flow cells, pistons, seals, fittings, valves, burners, sample tubes, probe inserts, print heads, glass lined tubing, pipe and tube fittings, variable temperature dewars, transfer lines, flexible discs, magnetic tape cassettes, electron multipliers, filaments, vacuum gaskets, seats and all parts exposed to samples and mobile phases.
This **warranty shall be void** in the event of accident, abuse, alteration, misuse, neglect, breakage, improper operation or maintenance, unauthorized or improper modifications or tampering, use in an unsuitable physical environment, use with a marginal power supply or use with other inadequate facilities or utilities. Reasonable care must be used to avoid hazards.

This warranty is expressly in lieu of and excludes all other express or implied warranties, including but not limited to warranties of merchantability and of fitness for particular purpose, use or application and all other obligations or liabilities on the part of Varian, unless such other warranties, obligations or liabilities are expressly agreed to in writing by Varian.

LIMITATION OF REMEDIES AND LIABILITY

The remedies provided herein are the sole and exclusive remedies of the Customer. In no case will Varian be liable for incidental or consequential damages, loss of use, loss of production or any other loss incurred.

SAFETY INFORMATION

For Safety Information in German, French, Italian or Spanish please refer to the 430-GC, 431-GC and 450-GC User Manual CD-rom (CP501414) which is included with the GC.

INFORMATION

To prevent any injury to the user or any damage to the instrument it is essential that you read the information in this chapter and the GC user manual.

If this manual is not in your native language and if you have problems understanding the text, we advise you to contact your Varian office for assistance. Varian cannot accept responsibility for any damage or injury caused by misunderstanding of the information in this manual.

OPERATING INSTRUCTIONS

This user manual is provided to help you establish operating conditions, which will permit safe and efficient use of your equipment.

Special considerations and precautions are described in the manual, which appear in the form of **NOTES**, **CAUTIONS** and **WARNINGS** as described on the next page.

It is important that you operate your equipment in accordance with this user manual and any additional information, which may be provided by Varian. Address any questions regarding the safe and proper use of your equipment to your local Varian office.

Information to aid you in obtaining optimal performance from your instrument.

Alerts you to situations that may cause moderate injury and/or equipment damage and how to avoid these situations.

Alerts you to potentially hazardous situations that could result in serious injury and how to avoid these situations.

Warning Symbol	Warning Description
	Hazardous voltages are present inside instrument. Disconnect from main power before removing screw-attached panels.
	Hazardous chemicals may be present. Avoid contact, especially when replenishing reservoirs. Use proper eye and skin protection.
	Very hot or cryogenically cold surfaces may be exposed. Use proper skin protection.
	Eye damage could occur either from flying particles, chemicals, or UV radiation. Use proper eye and face protection.
	The potential for fire may be present. Follow manual instructions for safe operation.
	The potential for explosion may exist because of type of gas or liquid used.
	Ionizing radiation source is present. Follow manual instructions for safe operation.
	Keep hands and fingers away.

GENERAL SAFETY PRECAUTIONS

Follow these safety practices to ensure safe equipment operation.

- Perform periodic leak checks on all supply lines and pneumatic plumbing.
- Do not allow gas lines to become kinked or punctured. Place lines away from foot traffic and extreme heat or cold.
- Store organic solvents in fireproof, vented and clearly labeled cabinets so they are easily identified as toxic and/or flammable materials.
- Do not accumulate waste solvents. Dispose of such materials through a regulated disposal program and not through municipal sewage lines.

This instrument has been tested per applicable requirements of EMC Directive as required to carry the European Union CE Mark.

CAUTION

This instrument is designed for chromatographic analysis of appropriately prepared samples. It must be operated using appropriate gases and/or solvents and within specified maximum ranges for pressure, flows and temperatures as described in this manual. If the equipment is used in a manner not specified by the manufacturer, the protection provided by the equipment may be impaired.

WARNING

It is the responsibility of the Customer to inform Varian Customer Support Representatives if the instrument has been used for the analysis of hazardous biological, radioactive, or toxic samples, prior to any instrument service being performed or when an instrument is being returned to the Service Center for repair.

ELECTRICAL HAZARDS

- Disconnect the instrument from all power sources before removing protective panels to avoid exposure to potentially dangerous voltages.
- When it is necessary to use a non-original power cord plug, make sure the replacement cord adheres to the color coding and polarity described in the pre-installation manual (P/N: CP01410) all local building safety codes.
- Replace blown fuses with fuses of the size and rating stipulated on the fuse panel or in the manual on page 44.
- Make sure that voltage sources and line voltage match the value for which the instrument is wired.
- Replace faulty or frayed power cords immediately with the same type and rating.

COMPRESSED GAS CYLINDERS

- Store and handle compressed gases carefully and in strict adherence to safety codes.
- Secure cylinders to an immovable structure or wall.
- Store and move cylinders in an upright, vertical position. Before transport, remove regulators and install cylinder cap.
- Store cylinders in a well-ventilated area away from heat, direct sunshine, freezing temperatures and ignition sources.
- Mark cylinders clearly so there is no doubt as to their contents.
- Use only approved regulators and connections.
- Use only connector tubing that is chromatographically clean (Varian Part Number 03-918326-00) and has a pressure rating significantly greater than the highest outlet pressure from the regulator.

GC SAFETY PRACTICES

Exhaust System

No special exhaust ducting is necessary for GC detectors installed in a well-ventilated room except when the detectors are used to test hazardous chemicals. If you do install ducting:

- Use only fireproof ducting.
- Install a blower at the duct outlet.
- Secure air duct intakes such that their vibration or air movement does not effect detector operation.
- Check periodically for proper operation of the duct.
- Ensure proper ventilation in lab area.

Radioactive Source Detectors

Read carefully and comply with all NOTES, CAUTIONS and WARNINGS in the Ni^{63} ECD section on page 329 of this manual.
Perform the tests for removable radioactive contamination described in the Ni^{63} ECD radiation manual (P/N: 03-913999-00). Comply with leak test schedules and procedures.

Burn Hazard

Heated or cryogenically cooled zones of the GC can remain hot or cold for a considerable time after instrument power is turned off. To prevent painful burns, ensure that all heated or cooled areas have returned to room temperature or wear adequate hand protection before you touch potentially hot or cold surfaces.

SPARE PARTS AVAILABILITY

It is the policy of Varian to provide operational spare parts for any instrument and major accessory for a period of five (5) years after shipment of the final production run of that instrument. Spare parts will be available after this five (5) year period but on an *as available* basis. Operational spare parts are defined as those individual electrical or mechanical parts that are susceptible to failure during their normal operation. Examples include relays, lamps, temperature probes, detector elements, motors, etc. Sheet metal parts, structural members or assemblies and castings, printed circuit boards and functional modules are normally capable of being rebuilt to like-new condition throughout their useful life and therefore will be supplied only on an *as available* basis after the final production run of the instrument.

SERVICE AVAILABILITY

Varian provides a variety of services to support its customers after warranty expiration. Repair service can be provided by attractively priced service contracts or on a time and material basis. Technical support and training can be provided by qualified personnel on both a contractual or as-needed basis.

Varian Analytical Instruments Sales Offices

For Sales or Service assistance and to order Parts and Supplies, contact your local Varian office. For the latest information visit www.varianinc.com.

Argentina
Buenos Aires
Tel. +54.11.4.783.5306

Australia
Mulgrave, Victoria
Tel. +61. 3.9560.7133

Austria
Poettelsdorf
Tel. +43.2626.20090

Benelux
Middelburg
Tel. +31.118.671500

Brazil and Latin America (S)
São Paulo
Tel. +55.11.32380400

Canada
Mississauga, Ontario
Tel. 800.387.2216

China
Beijing
Tel. +86.106310.8550

Europe
Middelburg, The Netherlands
Tel. +31.118.671.000

France
Les Ulis Cédex
Tel. +33.1.6986.3838

Germany
Darmstadt
Tel. +49.6151.7030

India
Mumbai
Tel.
+91.22.2570.8595/97

Italy
Torino
Tel. +39.011.997.9111

Japan
Tokyo
Tel. +81.3.5232.1239

Korea
Seoul
Tel. +82.333.665.5171

Mexico and Latin America (N)
Mexico City
Tel.
+52.5.55.5239465/026

Russian Federation
Moscow
Tel. +7.095.937.4280

Spain
Madrid
Tel. +34.91.472.7612

Sweden
Solna
Tel. +46.8.445.1620

Switzerland
Steinhausen
Tel. +41.848.803.800

Taiwan
Shih-Chi
Tel. +886.22.698.9555

United Kingdom and Ireland
Oxford
Tel. +44.1865.291500

Venezuela
Caracas
Tel.
+58.212.285.0320/2494

United States

Walnut Creek, California, USA
Tel. +1.800.926.3000
(GC and GC/MS)

Tel. +1.800.367.4752 (LC)

VARIAN

http://www.varianinc.com/

CE DECLARATION OF CONFORMITY

CE

Declaration of Conformity

We hereby Declare that the equipment listed below complies with the requirements of:
The Low Voltage Directive 73/23/EEC (93/68/EEC)
The EMC Directive 89/336/EEC (92/31/EEC and 93/68/EEC)

Applicable Standards

LVD	UL 61010-1 :2004 R7.05 CSA 22.2. 61010.1 :2004
EMC	EN 61326 :2003 47CFR part 15 J55022 :2002 Class B CISPR 22 :1996 Denan Law 1.6

Type of Equipment: Gas-Chromatograph **Model:** 450-GC

Manufacturer - EU

Print Name: G. A. Wassink
Signed: ___
Position: Quality Manager
Date: June 11, 2007

Company Name: Varian B.V.
Address: Herculesweg 8
P.O. Box 8033
4330 EA Middelburg
The Netherlands
Telephone: +31(0) 118 671 000
Fax: +31(0) 118 623 193

Manufacturer / Authorized Representative - USA

Print Name: Seamus Flanagan
Signed: Seamus Flanagan
Position: General Manager
Date: June 11, 2007

Company Name: Varian, Inc.
Address: 2700 Mitchell Drive
Walnut Creek, California 94598
USA
Telephone: 925-939-2400
Fax: 925-945-2360

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www.varianinc.com

TABLE OF CONTENTS

VARIAN ANALYTICAL INSTRUMENT WARRANTY ..... **III**

HARDWARE PRODUCTS III
SOFTWARE PRODUCTS III
REMEDIES III
LIMITATION OF WARRANTY III
LIMITATION OF REMEDIES AND LIABILITY III

SAFETY INFORMATION ..... **IV**

INFORMATION IV
OPERATING INSTRUCTIONS IV
GENERAL SAFETY PRECAUTIONS VI
ELECTRICAL HAZARDS VII
COMPRESSED GAS CYLINDERS VII
GC SAFETY PRACTICES VIII
Exhaust System ..... *VIII*
Radioactive Source Detectors ..... *VIII*
Burn Hazard ..... *VIII*
SPARE PARTS AVAILABILITY IX
SERVICE AVAILABILITY IX
CE DECLARATION OF CONFORMITY X

TABLE OF CONTENTS ..... **XI**

FIGURES ..... **XXV**

TABLES ..... **XXVII**

INTRODUCTION **1**

GUIDE TO THE 450-GC USER MANUAL 1
Abbreviations *1*
BRIEF DESCRIPTION OF THE 450-GC 2
CONFIGURATION AND OPTIONS 3

INSTALLATION **5**

NOTES ABOUT SETTING UP AND USING YOUR 450-GC 5
PREPARING FOR INSTALLATION 5
Inspection *5*
Unpacking *5*
Remove Protective Devices *6*
Connect Data Handling Peripherals *6*
Select Gases *7*
Hydrogen as Carrier Gas *8*

General laboratory precautions 9
Operating precautions 9
Install Gas Regulators and Set Pressures *10*
Connect Gases *10*
Installing the CP-8400/8410 AutoSampler *11*
Connect GC to Power 12
COLUMN INSTALLATION AND CONDITIONING 13
Nuts and Ferrules for Capillary Columns *14*
Connect Capillary Column to Injector *15*
Tips for Tightening Column Ferrules 17
Nuts and Ferrules for Packed Columns *18*
Connect a Packed Column to the Injector *18*
Varian Test Column *19*
Identify Carrier Gas Controls and Turn on Flow *19*
Split Vent Flow and Septum Purge Flow *20*
Septum purge calibration 20
Set Carrier Gas Flow Rate to Condition Column *22*
Injector Conditioning *22*
Column Conditioning *23*
New Columns 23
Used Columns 23
Connect Capillary Column to Detector *24*
Column Insertion Depths for Varian Detectors *24*
Connect Metal Packed Column to Detector *25*
Check for Leaks *25*
BASIC OPERATION 26
Setting Carrier and Detector Gas Flow Rates *26*
Condition the GC System *28*
Build a Method to Analyze a Test Sample *28*
Method to Analyze a Varian Test Sample on a Varian Test Column *29*
Prepare the Test Sample *30*
Solvents for GC *31*
Sample Volume and Injection Rate *32*
Inject the Test Sample *32*

REFERENCE 33

PERIPHERAL CONNECTORS 33

PERIPHERAL CONNECTORS 34
J504/J505 S.I.D. 1 AND 2 35
J401 ANALOG OUT 36
J302 TEMPERATURE PROBES 37
J45/J46 AUXILIARY 1 AND 2 38
J44 CRYO VALVES (3) 39
J42/J43 EXTERNAL EVENTS 40
J41 HEATER CONNECTIONS 41
REAR PANEL 42
OPTION BOARD 43
FUSES COMMON 44

Fuses *44*

DISPLAY AND KEYBOARD **45**

STATUS INDICATION LED'S 46
Overview Analytical Phases *46*
Error LED *47*
MOST USED FUNCTIONS 48
Status page *49*
Version Information *50*
ADVANCED FUNCTIONS 51
DISPLAY 52
Title Bar *53*
Main Operation screen *54*
Sample Delivery Screen *55*
Injector Screen *57*
Pressure and Flow screen *58*
Oven Screen *60*
Methods Screen *61*
Detector Screen *62*
Analog Output 63
Setup/Calibration *64*
Calibration 64
Setup 65
Configuration Tab 66
External Device 67
Sample Delivery Tab 68
Oven Tab 73
Column Tab 74
Detector Tab 75
Injector Tab 77
Preferences Tab 78
System Tab 79
Touch Screen Calibration 81
Error/Log Handling *84*

LOCAL AUTOMATION **87**

COMMUNICATIONS **89**

Introduction *89*
Ethernet Communications *89*
How to set IP address in Galaxie Workstation using BOOTP (DHCP) 90
How to set a Fixed IP address in the 450-GC 93
ANALOG CABLES 94

SAMPLE INTRODUCTION **97**

AUTOSAMPLERS 97
LIQUID INJECTORS 97
SAMPLING VALVES 99
Heated Zones for Valves 100

External Sample Introduction Systems *101*
Pneumatic Systems *101*

CP-8400/8410 AUTOSAMPLER 103

INTRODUCTION 103
INSTALLING THE CP-8400/8410 105
SAMPLER FIRST START UP 110
LARGE SOLVENT / WASH VIAL 111
CALIBRATION 112
Vial calibration *113*
GC Injector calibration *116*
SYRINGE CHECK/REPLACE 119
SYRINGE SIZE CHANGE 120
OPERATION 121
PARAMETERS AND QUICK REFERENCE 121
SAMPLER CONFIGURATION 122
SAMPLE DELIVERY PARAMETERS 123
Standard On-Column and Standard Split/Splitless Sampling Modes *124*
Neat Sampling Mode *126*
Viscous Sampling Mode *128*
Volatile Sampling Mode *129*
User Defined Sampling Mode *131*
Page 1 131
Page 2 135
Page 3 138
SPME Sampling Mode *140*
INSTALLATION OF THE SPME MODE 142
Injector Insert *145*
Injector Temperature *146*
Sample Vials and Septa *146*
Agitation Strokes *146*
Sample Penetration Depth (%) 147
SPME Adsorption Time 147
Number of Agitation Strokes 148
INSTALLATION OF THE LIQUID SAMPLING MODE 149

THE 1177 SPLIT/SPLITLESS CAPILLARY INJECTOR 153

Features of the 1177 Injector *154*
Automatic Start Switch *154*
1177 Injector Inserts *154*
1177 Modes of Injection *155*
Split Mode 155
Splitless Mode 155
1177 Injector Screen *156*
1177 Electronic Flow Control (EFC) *157*
1177 Manual Pneumatics *160*
Operation of the 1177 Injector *160*
Column Installation 160
Condition the Column 160

Column Installation in Detector 160
Setting 1177 Gas Flow Rates *161*
EFC Pneumatics *161*
Constant Column Flow *162*
Pressure Pulse *163*
Gas Saver *164*
Septum Purge Calibration *165*
Manual Pneumatics *166*
Tools and equipment needed 166
1177 Modes of Operation *167*
Split Injection 167
Splitless Injection *168*
Testing the 1177 Injector Performance *169*
1177 Injector Assembly *170*
Maintenance *171*
Tools Required 171
Remove the Glass Insert *171*
Replace the Glass Insert *172*
Injector body cleaning *173*

THE 1079 PTV INJECTOR 175

Features of the 1079 PTV Injector *176*
Automatic Start Switch *176*
1079 PTV Injector Inserts *176*
1079 PTV Injector Assembly *177*
1079 PTV Modes of Injection *178*
Temperature Ramp Splitless Mode 178
Large Volume 178
Split Mode 179
Isothermal Splitless Mode 179
On-Column Modes 179
1079 Injector Screen *180*
1079 PTV Electronic Flow Control (EFC) *181*
1079 PTV Manual Pneumatics *183*
Operation of the 1079 PTV Injector *183*
Column Installation 183
Condition the Column 183
Column Installation in Detector 183
Setting 1079 PTV Gas Flow Rates *184*
EFC Pneumatics *184*
Constant Column Flow 185
Pressure Pulse *186*
Gas Saver *187*
Septum Purge Calibration *188*
Manual Pneumatics *189*
Tools and equipment needed 189
1079 PTV Modes of Operation *190*
Isothermal Split Injection 190
Isothermal Splitless Injection 191

Temperature Ramped Splitless Injection 191
On-column Injection 192
Large Volume Injection 193
Testing the 1079 PTV Injector Performance 194
Maintenance 195
Tools Required 195
Remove the Glass Insert 195
Replace the Glass Insert 197
CHROMATOPROBE 198
How to Assemble the ChromatoProbe 198
How to Install the ChromatoProbe 198
Using the ChromatoProbe 200
How to Perform Analysis with the ChromatoProbe 200
Analysis of Thermally Labile Samples 201
Using the ChromatoProbe Along with Traditional GC/MS 202
Using the ChromatoProbe as a Dirty Sample Inlet 202
Cleaning 204
Recommended Reading 204

THE 1041 ON-COLUMN INJECTOR 205

Automatic Start Switch 206
1041 Injector Insert 206
Injector Assembly and Insert 207
1041 Electronic Flow Control (EFC) 208
Column Installation 211
Condition the Column 211
Column Installation in Detector 211
Setting 1041 Gas Flow Rates 211
EFC Pneumatics 211
Flow Rates for Operation 212
Capillary Mode 212
Packed Mode 212
1041 Operation 213
1041 Injector Method 213
Testing the 1041 Injector 214
Maintenance 215
Hardware Replacement and Cleaning Procedures 215
Septum Replacement 215
Needle/ Syringe Cleaning 216
Leak Checking 216
Syringe Leak Checking 217

THE 1061 FLASH VAPORIZATION INJECTOR 219

Automatic Start Switch 220
Injector Assembly and Insert 221
1061 Electronic Flow Control (EFC) 222
1061 Injector Insert 224
Column Installation 224
Column Installation 224

Condition the Column 224
Column Installation in Detector 224
Setting 1061 Gas Flow Rates *225*
EFC Pneumatics *225*
Flow Rates for Operation 226
Capillary Mode 226
Packed Mode 226
1061 Flash Injector Operation *227*
1061 Flash Injector Method 227
Testing the 1061 Flash Injector *228*
MAINTENANCE 229
Replacing the 1061 Glass Insert *229*
Remove the Glass Insert *229*
Replace the Glass Insert *230*

1093 SEPTUM-EQUIPPED PROGRAMMABLE INJECTOR (SPI) 231

Automatic Start Switch *233*
Injector Assembly and Insert *234*
1093 Injector Parts *234*
1093 Electronic Flow Control (EFC) *235*
Glass Insert *237*
Syringe *237*
Mounting the Column *238*
Column Installation *238*
Condition the Column *238*
Column Installation in Detector *238*
Connecting CO_2 to the 1093 Injector *238*
Connecting LN2 to the 1093 Injector *239*
Setting 1093 Gas Flow Rates *240*
EFC Pneumatics *241*
1093 Injector Operation *242*
Testing the 1093 Injector Performance *243*
Septum Purge Calibration *244*
Maintenance *245*
Septum Replacement *245*
Removal of Glass Insert 245
Removal 245
Re-assemble 246
Leak Checking *247*
Leak Checking the Injector Only 247
Gas Leak Checking 247
Visual Leak Checking 248
Syringe Leak Checking 248
Injector and Column Conditioning *248*
Baking Out the Injector 248

SAMPLE PRECONCENTRATION TRAP (SPT) 251

Description *251*
SPT temperature zone *252*

Two trap sizes available *253*
Using the SPT *253*
Trapping Methodology 253
Trapping Media 253
Trap Dimensions 254
Trap Temperature 254
Trapping and Desorption 254
Electrical System *255*
Circuit breaker 256
LCO_2 Option *257*
System Description 257
Theory of Operation 258
Characteristics of LCO_2 as a Coolant 258
Inadequate Cooling 258
Transfer Line 259
Recommended Supply Conditions 259
LN_2 Option *260*
Description 260
Recommended Supply Conditions 261
Recommended temperature ranges 261
Configuring the SPT *262*
Building an SPT method *263*
Replacing the Trap Assembly *264*
SPT System Components *265*
Remove the SPT Cover *266*
Detach Trap Assembly *267*
Remove Cryo Nozzle Assembly *268*
Installing the Trap Assembly *269*
Trap Conditioning *273*
Replacement SPT traps *274*
SPT replacement parts *275*
Troubleshooting *276*

DETECTORS 279

INTRODUCTION 279
ANALOG OUTPUT 280

FLAME IONIZATION DETECTOR (FID) 283

Initial Set-Up *284*
Operation *285*
FID auto-Ignite flow diagram *287*
Installation/Disassembly *288*
Exploded View 288
Disassemble the FID 289
Reassemble the FID 290
MAINTENANCE 292
Clean the Flame Tip and Internal Parts *292*
Replace the Flame-Tip Ferrule *293*
FID Test Chromatogram *293*

PULSED DISCHARGE HELIUM IONIZATION DETECTOR (PDHID) 295

HELIUM IONIZATION (PDHID) 296
Cable overview 297
Exploded View 298
Gas Connections 299
Installation 299
General Precautions 300
Installing and Purging the Helium Purifier 300
Connecting the Discharge Gas to the Detector 301
Capillary Column Connection 301
Packed Column Connection 302
Testing for Leaks 303

THERMIONIC SPECIFIC DETECTOR (TSD) 305

Initial Set-Up 306
Operation and Operating Recommendations 307
Bead Conditioning 307
Optimization of the TSD Using the Varian Test Sample 309
Determining Optimum Hydrogen Flow 309
Maintaining Optimum Conditions 310
Column Bleed 311
Solvent Considerations 311
Chlorinated Solvents 311
Effect of Silylation Reagents 312
TSD Assembly 313
Exploded view 313
TSD disassembly 314
TSD Re-assembly 315
MAINTENANCE 317
Cleaning / Maintenance 317
Bead Cleaning 317
Flame Tip and Internal Parts Cleaning 317
Replace the Flame-Tip Ferrule 318
Test Chromatogram 318

THERMAL CONDUCTIVITY DETECTOR (TCD) 319

Before Operating the TCD 320
Initial Set-Up 320
TCD Adjustments 322
Operation 323
Range Setting 325
Low Currents (100-150 mA) 325
High Currents (300 mA) 326
Calculate Detector Sensitivity 327
Calculate Detector Detectivity 328
Adjust TCD Carrier Gas Flow Rates 328
MAINTENANCE 328
TCD Test Chromatogram 328

ELECTRON CAPTURE DETECTOR (ECD) 329

Initial Set-up 330
ECD Adjustments 331
Cell-Current 331
Time Constant Switch 331
Setting the Contact Potential 332
ECD Operation 333
Column and Septum Bleed 333
Column Conditioning 333
Column Oven and Detector Oven Temperature Selection 333
ECD Pneumatics 334
Installation/Disassembly 335
Exploded View 335
ECD Installation 337
MAINTENANCE 340
Thermal Cleaning With Carrier Gas 340

PULSED FLAME PHOTOMETRIC DETECTOR (PFPD) 343

Description 343
Basic Components of the PFPD 343
Features of the PFPD Assembly 344
Features of the PFPD Electrometer Board 346
Theory of Operation of the PFPD 346
Stages of Flame Pulsation 347
How Does the PFPD Achieve its Greater Detectivity and Selectivity? 348
Background Emission in a Pulsed-flame 348
Time-dependence of Emission Profiles 348
Emission Profiles 348
Concentration (in time) of the Emitted Light 349
Separation of the Element Emission from Background Emission 349
Effect of Gate Control 350
Adjustment of the Gas Composition 351
PFPD Flow Path 351
References 352
Specifications 352
Installation 354
Inspecting the PFPD 354
PFPD Accessory Kit 354
PFPD Diffusion Source Kit 355
Installing a Chromatographic Column 356
Prepare the Column End 356
Disassemble the PFPD 357
Remove the Detector Tower (Optional) 359
Insert the Column 360
Reassemble the PFPD 361
Operation 363
Adjusting the Gas Flow Rates 363
Split Valve 363
PFPD Gas Flow Diagram with EFC15 364

Adjust the Gas Flow Rates *365*
Calibrating the Flow Controllers *366*
Optimizing the PFPD *368*
Detectivity *368*
Selectivity *368*
Throughput *368*
Suggested Fuel Gas Flow Rates *369*
Optimization Criteria *370*
Establishing Tick-Tock *375*
Photomultiplier Tube (PMT) High Voltage *378*
Adjusting the Photomultiplier Tube (PMT) Voltage *378*
Optimizing the PFPD *379*
Repeated Injections of Standard *379*
Continuous Infusion of Standard *381*
Testing the PFPD Performance *382*
PFPD Test Chromatogram: Sulfur Performance *385*
PFPD Test Chromatogram: Phosphorus Performance *386*
Detectivity *386*
PFPD Test Chromatogram: Sulfur Detectivity *387*
PFPD Test Chromatogram: Phosphorus Detectivity *388*
Using the Gate Delay and Gate Width Parameters *388*
Setting the Trigger Level *390*
Setting Gate Delay and Gate Width *392*
Set Gate Width *393*
Building a GC Method *394*
Building a GC Workstation Method *395*
Quantitation *396*
Peak Area Quantitation *396*
Peak Height Quantitation *397*
Peak Resolution with the PFPD *397*
Quantitation in the Sulfur Mode *397*
Square Root Function *398*
How to Minimize Quenching of Sulfur Response *398*
Advanced Operation *399*
Theory of Operation *399*
Table of Optimum Flow Rates *400*
Installation of the Nitrogen Mode Parts *400*
Nitrogen Mode Operation *402*
Parts and Supplies (Nitrogen Mode) *403*
Phosphorus Mode Operation *403*
Sulfur Mode Operation *403*
Options to Aid Optimization with Other Elements *403*
Multi-element Detection *404*
Arsenic Selective Detection *404*
Emission Wavelengths *404*
Emission Timing *404*
Flame Composition *404*
Filters *405*
Selenium Selective Detection *405*

Emission Wavelengths *405*
Emission Timing *406*
Flame Composition *406*
Filters *406*
Tin Selective Detection *406*
Emission Wavelengths *406*
Emission Timing *407*
Flame Composition *407*
Filters *407*
Manganese Selective Detection *407*
Emission Wavelengths *408*
Emission Timing *408*
Flame Composition *408*
Filters *408*
Maintaining the PFPD *409*
Replacing the Optical Filter *409*
Changing the Combustor Size *411*
Replacing a Dirty Combustor *411*
Ordering Parts and Supplies for the PFPD *412*
Troubleshooting *414*
Overview *414*
Flame Does Not Ignite *414*
Baseline Noise is Too High *415*
Baseline is Erratic (tick-tock) *417*

DETECTOR TEST CHROMATOGRAMS **419**

FID Test Mix *420*
TSD Test Mix *421*
TCD Test Mix *422*
ECD Test Mix *423*
PFPD P Mode *424*
PFPD S Mode *425*

GENERAL GC MAINTENANCE **427**

Overview *427*
General GC Maintenance *428*
Check and Renew Gas Supplies 428
Leak Check *428*
Gas Purifier Replacement *430*
Injector Maintenance *432*
Septum Replacement 432
Instructions for High-Temperature bakeout of Injectors 433
Injector specified maintenance *436*
Detector specified maintenance *436*
Pencil Filter *437*

SHIPPING, CLEANING AND DISPOSAL INSTRUCTIONS **439**

SHIPPING INSTRUCTIONS 439
CLEANING INSTRUCTIONS 439

DISPOSAL INSTRUCTIONS 439

REPLACEMENT PARTS 441

Injector Liners/Inserts 443
Liner/Insert Descriptions 445

TECHNICAL TIPS 457

CHROMATOGRAPHIC INTERPRETATIONS 458
TO BLEED OR NOT TO BLEED … THAT IS THE QUESTION 461
The baseline below 250 °C 461
Injector contamination 462
The Column 463
Gas Impurities 465
CHOOSING THE RIGHT GC INJECTION TECHNIQUE 466
Direct Injection 467
Description 467
Split Injection 467
Description 467
Splitless Injection 468
Description 468
Cool on-column 470
Description 470
Programmed Temperature Vaporizer (PTV) 470
Description 470
Cold Split Injection 471
Cold Splitless Injection 471
Solvent Venting 471
GC LINER LABYRINTH - HOW TO CHOOSE THE RIGHT INJECTOR INSERT 472
Liner Types 472
Liner volume 473
Injector heat capacity 473
Discrimination effects 474
Liners for splitless injections 475
Straight liner 475
Gooseneck 475
Gooseneck with wool 475
Double Gooseneck 475
Recessed Gooseneck 476
Liners for split injections 476
Frit Splitter 476
Inverted -or Laminar Cup Splitter 476
Baffle Splitter 476
Liners for direct injections 477
Straight tube 477
Gooseneck 477

APPENDIX A: ERROR CODES 479

APPENDIX B: PUBLIC STATEMENT 491

FIGURES

FIGURE 1: COLUMN ENDS 15
FIGURE 2: CONNECTORS FOR PERIPHERAL EQUIPMENT 34
FIGURE 3: CONNECTOR J504-J505 35
FIGURE 4: CONNECTOR J401 36
FIGURE 5: CONNECTOR J302 37
FIGURE 6: CONNECTOR J45/J46 38
FIGURE 7: CONNECTOR J44 39
FIGURE 8: CONNECTOR J42/J43 40
FIGURE 9: CONNECTOR J41 41
FIGURE 10: OPTION BOARD 43
FIGURE 11: 450-GC BACK-PANEL ETHERNET CONNECTION 89
FIGURE 12: PNEUMATIC SYSTEMS FOR LIQUID INJECTORS AND SAMPLING VALVES 101
FIGURE 13: TOP-COVER FOR SAMPLER 105
FIGURE 14: SPME SYRINGE AND HOLDER FIGURE 15: SPME/STANDARD SWITCH 142
FIGURE 16: SPME SYRINGE 145
FIGURE 17: LIQUID SAMLING SYRINGE AND HOLDER FIGURE 18: SPME/STANDARD SWITCH 149
FIGURE 19: EFC21 AND 1177 IN SPLIT MODE FLOW DIAGRAM 157
FIGURE 20: EFC21 AND 1177 IN SPLITLESS MODE FLOW DIAGRAM 158
FIGURE 21: EFC25 AND 1177 FLOW DIAGRAM 159
FIGURE 22: 1177 INJECTOR ASSEMBLY 170
FIGURE 23: 1079 PTV INJECTOR ASSEMBLY 177
FIGURE 24: EFC21 AND 1079 IN SPLIT MODE FLOW DIAGRAM 181
FIGURE 25: EFC25 AND 1079 FLOW DIAGRAM 182
FIGURE 26: CHROMATOPROBE ASSEMBLY 199
FIGURE 27: 1041 ON-COLUMN INJECTOR WITH INSERT 207
FIGURE 28: EFC23 AND 1041 FLOW DIAGRAM 209
FIGURE 29: EFC24 AND 1041 FLOW DIAGRAM 210
FIGURE 30: 1061 FLASH VAPORIZATION INJECTOR WITH INSERT 221
FIGURE 31: EFC23 AND 1061 FLOW DIAGRAM 222
FIGURE 32: EFC24 AND 1061 FLOW DIAGRAM 223
FIGURE 33: 1093 INJECTOR ASSEMBLY 234
FIGURE 34: EFC23 AND 1093 FLOW DIAGRAM 235
FIGURE 35: EFC24 AND 1093 FLOW DIAGRAM 236
FIGURE 36: CONNECTION CO_2 TANK TO GC 238
FIGURE 37: SPT ELECTRICAL DIAGRAM 256
FIGURE 38: LCO_2 COOLANT FLOW DIAGRAM 257
FIGURE 39: LN_2 COOLANT FLOW DIAGRAM 260
FIGURE 40: SPT COMPONENTS (SHORT TRAP ASSEMBLY, LCO_2 OPTION SHOWN 265
FIGURE 41: REMOVING SPRINGS FROM TRAP ASSEMBLY 267
FIGURE 42: CRYO NOZZLE ASSEMBLY 268
FIGURE 43: FID AUTO-IGNITE FLOW DIAGRAM 287
FIGURE 44: FID EXPLODED VIEW 288
FIGURE 45: ORIENTATION OF IGNITOR PROBE ELEMENTS IN FID TOWER 291
FIGURE 46: FID CROSS-SECTIONAL VIEW 291

FIGURE 47: SCHEMATIC OF THE PDHID DETECTOR 296
FIGURE 48: PDHID CABLE OVERVIEW..... 297
FIGURE 49: PDHID EXPLODED VIEW..... 298
FIGURE 50: PDHID GAS CONNECTIONS OVERVIEW 299
FIGURE 51: STRUCTURE OF COMPONENTS IN THE TSD TEST SAMPLE & CHROMATOGRAM EXAMPLES 310
FIGURE 52: TSD EXPLODED VIEW..... 313
FIGURE 53: TSD CROSS-SECTIONAL VIEW 316
FIGURE 54: TCD SENSITIVITY AND AMPLIFICATION..... 326
FIGURE 55: ECD EXPLODED VIEW 335
FIGURE 56: RADIOACTIVE CAUTION LABEL – GENERAL LICENSE DEVICE 338
FIGURE 57: RADIOACTIVE CAUTION LABEL – SPECIFIC LICENSE..... 338
FIGURE 58: RADIOACTIVE CAUTION LABEL 339
FIGURE 59: PFPD ASSEMBLY 344
FIGURE 60: FID TEST MIX CHROMATOGRAM..... 420
FIGURE 61: TSD TEST MIX CHROMATOGRAM 421
FIGURE 62: TCD TEST MIX CHROMATOGRAM 422
FIGURE 63: ECD TEST MIX CHROMATOGRAM..... 423
FIGURE 64: PFPD P MODE TEST MIX CHROMATOGRAM..... 424
FIGURE 65: PFPD S MODE TEST MIX CHROMATOGRAM..... 425

TABLES

TABLE 1: HYDROGEN AS CARRIER GAS DURING POWER OFF 8
TABLE 2: GAS INLET PRESSURE 10
TABLE 3: FACTORY-INSTALLED INJECTOR INSERTS 13
TABLE 4: 1/16” COLUMN FERRULES 14
TABLE 5: MAXIMUM INSERT DEPTH FOR CAPILLARY COLUMN IN INJECTOR 16
TABLE 6: GENERAL TIGHTENING AND RE-TIGHTENING FOR COMMON FERRULES 17
TABLE 7: VARIAN TEST COLUMNS 19
TABLE 8: INJECTOR AND EFC TYPE COMBINATION 19
TABLE 9: COLUMN CONDITION HEAD PRESSURE 22
TABLE 10: FLOW RATES FOR CARRIER AND DETECTOR GASES 26
TABLE 11: DETECTOR MANUAL FLOW RATES 27
TABLE 12: VARIAN TEST SAMPLES 29
TABLE 13: VARIAN TEST SAMPLE 1041 AND 1061 CONDITIONS 29
TABLE 14: VARIAN TEST SAMPLE 1177 AND 1079 CONDITIONS 30
TABLE 15: RECOMMENDED SOLVENTS FOR GAS CHROMATOGRAPHY 31
TABLE 16: USED FUSES 44
TABLE 17: STATUS INDICATION LED'S 46
TABLE 18: ERROR LED 47
TABLE 19: INJECTOR EFC MODULES 59
TABLE 20: DETECTOR EFC MODULES 63
TABLE 21: SAMPLER/INJECTOR POSITION 69
TABLE 22: VALVE TYPE NAMES 72
TABLE 23: ANALOG CABLES 95
TABLE 24: INJECTION MODE PARAMETERS FOR ON-COLUMN 125
TABLE 25: INJECTION MODE PARAMETERS FOR SPLIT-/SPLITLESS 126
TABLE 26: INJECTION MODE PARAMETERS FOR NEAT 128
TABLE 27: INJECTION MODE PARAMETERS FOR VISCOUS 129
TABLE 28: INJECTION MODE PARAMETERS FOR VOLATILE 130
TABLE 29: SPLIT INJECTION TYPICAL CONDITIONS 167
TABLE 30: SPLITLESS INJECTION METHOD PARAMETERS 168
TABLE 31: DETECTOR TEST SAMPLES 169
TABLE 32: 1177 COLUMN FERRULES 1/16" 170
TABLE 33: 1079 COLUMN FERRULES 1/16" 177
TABLE 34: SPLIT INJECTION TYPICAL CONDITIONS 190
TABLE 35: ISOTHERMAL SPLITLESS INJECTION METHOD PARAMETERS 191
TABLE 36: TEMPERATURE RAMPED SPLITLESS INJECTION 192
TABLE 37: ON-COLUMN INJECTION 193
TABLE 38: LARGE VOLUME INJECTION 194
TABLE 39: 1079 PTV TEST SAMPLES 194
TABLE 40: 1041 COLUMN FERRULES 1/16" X 0.8 MM 207
TABLE 41: CARRIER GAS FLOW RATES FOR 1041 212
TABLE 42: 1041 INJECTOR CHROMATOGRAPHIC TESTING CONDITIONS 214
TABLE 43: 1061 COLUMN FERRULES 1/16" X 0.8 MM 221
TABLE 44: CARRIER GAS FLOW RATES FOR 1061 FLASH INJECTOR 226

TABLE 45: 1061 FLASH INJECTOR CHROMATOGRAPHIC TESTING CONDITIONS 228
TABLE 46: DETECTOR TEST SAMPLES 243
TABLE 47: DETECTABLE GAS LEAK CHECKING 247
TABLE 48: SPT RECOMMENDED OPERATING RANGES 261
TABLE 49: SPT TRAP CONDITIONING 273
TABLE 50: SPT REPLACEMENT TRAPS 274
TABLE 51: SPT REPLACEMENT PARTS 275
TABLE 52: ECD GASES, PRESSURES, PURITY AND FLOW RATES 334
TABLE 53: SPECIFICATIONS PFPD 352
TABLE 54: SPECIFICATIONS NITROGEN MODE PFPD 353
TABLE 55: ACCESSORY KIT PFPD 354
TABLE 56: DIFFUSION SOURCE KIT PFPD 355
TABLE 57: FLOW RATES PFPD 369
TABLE 58: PRESET VALUES PFPD 389
TABLE 59: RECOMMENDED SETTINGS FOR COMMON ELEMENTS 392
TABLE 60: OPTIMUM FLOW RATES PFPD 400
TABLE 61: DETECTOR TEST SAMPLES 419
TABLE 62: MAINTENANCE FREQUENCY 427
TABLE 63: VARIAN GAS CLEAN FILTERS 431
TABLE 64: INJECTOR BAKEOUT PARTS 433

INTRODUCTION

GUIDE TO THE 450-GC USER MANUAL

The purpose of this User Manual is to provide instructions for the safe installation and operation of the Varian 450-GC (Gas Chromatograph).

Abbreviations

The following abbreviations can appear in this Manual:

AC	alternating current
DC	direct current
ECD	Electron Capture Detector
EFC	Electronic Flow Control
FID	Flame Ionization Detector
PFPD	Pulsed Flame Photometric Detector
TCD	Thermal Conductivity Detector
TSD	Thermionic Specific Detector
GC	Gas Chromatograph
GC/MS	Gas Chromatograph/Mass Spectrometer
ID	Inner Diameter
OD	Outer Diameter
P/N	Part Number
LUI	Local User Interface
S.I.D.	Sample Introduction Device connector
kPa	Kilo Pascal gauge
psi	Pounds per squari inch gauge

BRIEF DESCRIPTION OF THE 450-GC

The following are some of the key features of the 450-GC:

- Injectors, columns and detectors can be installed and operated simultaneously.
- The 1079 PTV Injector provides 5 modes of injection - isothermal split and splitless, temperature ramped splitless, on-column and large volume.
- Electronic or manual control of carrier and detector gas.
- Ethernet® communications with full networking capability.
- A full suite of universal and selective detectors - FID, TCD, ECD, TSD (N, P), PFPD (S, P, N, C, Metals), PDHID and Ion Trap MS.
- Large, swing-out pneumatics for easy access.
- 7 heated zones and 7 external events (24 Volt) for maximum flexibility and custom configurations.
- Option board with 8 external events (24 Volt) available, also 3 analog outputs with output voltage 0-1 and 0-10 Volt.
- Up to 99 separately programmed and stored analytical methods.
- Intuitive, function-based keyboard, LCD display with touch-screen for ease of use in instrument handling.
- Built-in Custom Solutions capability - valved systems, custom plumbing, six-position valve oven, methanizer.
- Large column oven for easy access and installation of up to three analytical columns.
- A complete range of GC automation and sample preparation techniques - , the CP-84X0 AutoSamplers, Solid Phase MicroExtraction, Purge and Trap, Heated Headspace and the Archon Purge and Trap AutoSampler.

CONFIGURATION AND OPTIONS

The 450-GC has seven heated zones, one of which is always configured as the column oven. The following options may be configured in the other 6 available heated zones:

- 1177 Split/Splitless Capillary Injector.
- 1079 PTV Injector.
- 1041 On-Column Injector for wide bore (0.53 mm ID) or packed columns.
- 1061 Flash Vaporization Injector for wide bore or packed columns.
- Single, dual, or multi-position Valve Oven.
- Flame Ionization Detector (FID).
- Pulsed Discharge Helium Ionization Detector (PDHID).
- Thermal Conductivity Detector (TCD).
- Electron Capture Detector (ECD).
- Thermionic Specific Detector (TSD).
- Pulsed Flame Photometric Detector (PFPD).
- Methanizer.

Any combination of three Injectors may be installed on the 450-GC. If a valve oven is installed, it normally replaces 2 physical Injector positions.

Any combination of three detectors can be installed with the following exceptions: Only 2 TCD or PFPD detectors can be installed. If 2 standard TCDs are installed, no other detectors can be accommodated (physical space). However, a dual TCD (two cells in one housing) is available as a Custom Solution which allows installation of another detector, such as an FID, behind it.

The methanizer typically occupies the same location as one of the detectors.

Installed options on the 450-GC are generally identified by their location. The options mounted on the top of the instrument, such as those listed above, are designated as Front, Middle and Rear to coincide with their relative location or the location of their respective electronic control modules.

INSTALLATION

NOTES ABOUT SETTING UP AND USING YOUR 450-GC

The purity of the gases used with your 450-GC are essential for optimum operation.
Varian Gas Clean Filter Kit (CP736530) is highly recommended.
All GCs require regular maintenance in order to operate at their highest efficiency.
You want to change the Injector septum, Injector liner, FID flame tip, gas filters and AutoSampler vials, wash bottle septa, syringes and solvents regularly.
Varian has an extensive supply of quality maintenance parts for your 450-GC and all of your chromatography needs including a comprehensive selection of the highest quality capillary columns available to chromatographers. Please visit the Varian internet site (www.varianinc.com), contact your Varian sales representative, or call your local Varian distribution center.

PREPARING FOR INSTALLATION

CAUTION

Refer to the Pre-Installation Instructions (CP501410) for site preparation and unpacking information.

Inspection

The GC will arrive packed in one large palletized box and one or more smaller cartons. Inspect the cartons carefully for damage or signs of rough handling. Report damage to the carrier and to your local Varian office.

Unpacking

Unpack the GC and accessories carefully and transfer to the work area, using proper handling techniques. The GC should be lifted from the bottom. Inspect the GC and accessories and immediately report any damage to the carrier and your local Varian office.

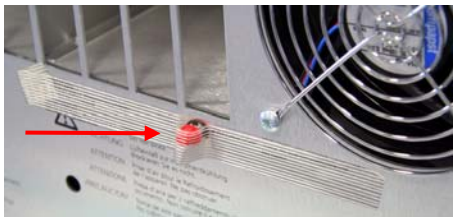
WARNING

Avoid back strain or injury by following all safety precautions when lifting heavy objects.

Remove Protective Devices

The instrument has been protected during shipment by various caps, plugs and restraints. Prior to operating, remove:

- Transport clamp for oven fan motor from hole in GC rear panel.
- Detector tower plugs under Detector cover.
- Gas bulkhead fitting plugs on rear panel.
- Any plastic plugs, tape or plastic material inside the column oven.



Connect Data Handling Peripherals

Workstations and other data handling devices are connected to the cable connectors on the back panel or the top of the instrument.

- The Varian Workstation of Galaxie with 450-GC control must be installed. The hardware installation simply involves making a data-connection, on page 89, from the computer (or hub) to the Ethernet® connector on the rear panel of the GC on page 42.
- If an analog output signal (optional) is being used, follow the specific installation instructions provided with the analog interface kit.

Select Gases

The following gases are commonly used in Gas Chromatography for the following functions. Note that the recommended purity for carrier make-up and detector fuel gases is 99.999%. In addition, the use of gas filters to remove hydrocarbons, oxygen and water from carrier gases is recommended.

Gas	Function
He, N_2, H_2, Ar, Ar/CH_4	Carrier gas
H_2, Air	Detector fuel gases
He, N_2, Ar/CH_4	Make-up gas for capillary column use
Air	AutoSampler operation
LN_2, LCO_2	Cryogenics

WARNING

Do not exceed MAXIMUM inlet pressures listed on the rear panel of the instrument.

Hydrogen as Carrier Gas

The use of hydrogen as a GC carrier gas is potentially dangerous.

Hydrogen (H_2) is a commonly used GC carrier gas. Hydrogen, however, is potentially explosive and has other characteristics that make it dangerous.

- Hydrogen is combustible over a wide range of concentrations. At atmospheric pressure, hydrogen is combustible at concentrations from 4% to 74.2% by volume.
- Hydrogen has the highest burning velocity of any gas.
- Hydrogen has very low ignition energy.
- Hydrogen temperature increases as the gas expands.
- Hydrogen that is allowed to expand rapidly from high pressure can self ignites.
- Hydrogen burns with a no luminous flame which can be invisible under bright light.

WARNING

Hydrogen is potentially explosive. Take extreme care when using hydrogen as the GC carrier gas in a GC system.

All users should be aware of the mechanisms by which hydrogen can accumulate. They should also know what precautions to take if they know, or suspect, that hydrogen has accumulated.

Mechanism	*Results*
GC off	A GC can be shut down deliberately. It can also be shut down accidentally by an internal or external failure. If a GC equipped with Electronic Flow Control (EFC) is shut off, the EFC does stop the flow of carrier gas.
Power failure	If the power fails, the GC shuts down. The carrier gas is shut down (in case of EFC).

Table 1: Hydrogen as Carrier Gas during Power off

General laboratory precautions

- Avoid leaks in the carrier gas lines. Use leak-checking equipment to periodically check for hydrogen leaks.
- Eliminate from your laboratory as many ignition sources as possible (open flames, devices that can spark, sources of static electricity, etc.).
- Do not allow hydrogen from a high pressure cylinder to vent directly to atmosphere (danger of self ignition).
- Use a hydrogen generator instead of bottled hydrogen.

Operating precautions

- Turn off the hydrogen at its source any time you shut down the GC.
- Turn off the hydrogen at its source if a power failure occurs.

Install Gas Regulators and Set Pressures

Carrier gas, air and H_2 supplied from cylinders or manifolds should have a two-stage regulator having a 0 to100 psi low-pressure stage. Set cylinder regulator pressure to match the GC gas inlet pressures, listed below:

Gas Inlet	*Pressure*
Carrier gas	500 kPa, 5 Bar, 80 psi (maximum 1000 kPa/ 10 Bar / 150 psi)
Make-up	500 kPa, 5 Bar, 80 psi (maximum 1000 kPa/ 10 Bar / 150 psi)
Air	400 kPa, 4 Bar, 60 psi (maximum 1000 kPa/ 10 Bar / 150 psi)
H_2	300 kPa, 3 Bar, 40 psi (maximum 650 kPa/ 6.5 Bar / 100 psi)

Table 2: Gas inlet pressure

Calculate a GC pressure drop (restrictions, tubing and EFC) of about 200 kPa, 2 Bar, 30 psi for well working EFC's.
Example: Column pressure needed 100 kPa + 200 kPa (pressure drop) = Inlet pressure needed at least 100+200= 300kPa.

WARNING

Maximum Inlet Pressure:

Air/N_2/He/Ar: 1000 kPa / 10 Bar / 150 psi
H_2: 650 kPa / 6.5 Bar / 100 psi

Connect Gases

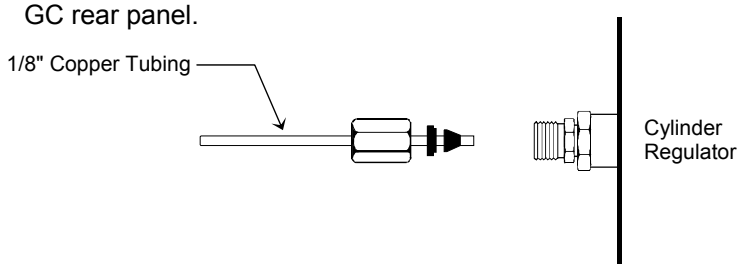
Follow the steps below to connect gas supplies to your GC:

1. Cut required lengths of chromatographically-cleaned 1/8″ copper tubing (P/N: 03-918326-00) for the carrier and fuel gas plumbing. Clean the ends of the tubing with a metal file.

2. Slide a 1/8″ Swagelok nut, back ferrule and front ferrule over one end of the copper tubing and attach to the outlet fitting on the cylinder regulator.

3. Push the copper tubing into the outlet fitting as far as it will go, then pull back very slightly and tighten 3/4-turn past finger-tight with a 7/16″ wrench.

4. Slide a 1/8″ Swagelok nut, back ferrule and front ferrule over the other end of the copper tubing and connect it to the corresponding gas inlet on the GC rear panel.



5. Push the copper tubing into the bulkhead fitting as far as it will go, then pull back very slightly and tighten 3/4-turn past finger-tight with a 7/16" wrench.

6. Make sure the gas supply control valve, which controls gas pressure to the GC, is completely closed. Open this valve slowly and monitor the pressure gauges on the dual stage regulator. The first pressure gauge should now read the pressure of the cylinder.

7. Cautiously turn the regulator valve to supply gas to the GC. Watch the pressure gauge closely and adjust the pressure according to Table 2 on page 10.

8. Leak checks all fittings.

Installing the CP-8400/8410 AutoSampler

To install your CP-8400/8410 on a 450-GC, first unpack the AutoSampler and verify that all of the parts required are present.
Inspect the GC and accessories and immediately report any damage to the carrier and your local Varian office.

For a detailed installation instructions please refer to the CP-8400 AutoSampler and CP-8410 AutoInjector section in this manual, on page 103.

It is important that the CP-8400/8410 is switched on prior to powering up the GC.
A flashing green LED at the rear of the AutoSampler is indicating that there is communication between GC and the Sampler.

Connect GC to Power

Plug the GC power-cord into an appropriate source of power.

Varian supplies a number of power-cords qualified to be used with the power load of the GC. Depending on the local electrical network different power-cords are available.

WARNING

Do not use power-cords with to low capacity. This may result in overheating of the power-cord.

Turn on the GC power switch located at the top left of the GC.

CAUTION

The GC top cover (power switch) of the GC must be free of any obstacles. This will give in case of emergency quick access to the power switch.

- The 450-GC automatically enters the OPERATION screen after power up.

  Press the SETUP key and select Configuration to verify the 450-GC configuration.

Your GC is shipped from the factory with default settings. The following is relevant information on the factory default states and settings:

- When the GC is turned the first time on, all installed heated zones except for the column oven default to power OFF.
- The column oven defaults to power ON and a set temperature of 50°C.
- When installed components that use a heated zone are turned ON in the active method, they default set to a temperature of 50°C.

COLUMN INSTALLATION AND CONDITIONING

Your GC arrives ready for installation of an analytical column. On first installation, a new column needs to be conditioned.

Conditioning is performed with the detector end of the column not connected.

WARNING:
BURN HAZARD

You may need to install and condition a new column after your GC has been in use for some time. Reset all Injector, detector and column ovens to ≤ 50°C to avoid burn hazards when connecting column to Injector and detector parts inside the oven. See also Column Conditioning on page 23.

Prior to installing your column, check that the factory-installed Injector is equipped with the proper insert for your application. Factory-installed Injectors, other than the 1041, have a glass insert suitable for a variety of applications. If your application calls for a different insert, replace the factory-installed insert prior to installing your column. Refer to the Injector section of the Manual for instructions to replace the insert on page 97.

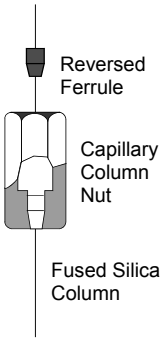
The factory-installed inserts for the Injectors are shown below, more Injector insert you can find here, on page 443.

Injector	*Insert Information*	*Part Number*
1061	Flash Injector Liner, 0.53 mm ID Capillary columns	03-92611943
1079 PTV	3.4 mm unpacked Injector insert	03-92611945
1093	0.8mm ID, 0.5mm restriction SPI deactivated Injector Liner	SG092034
1041	Insert for 0.53 mm ID columns	03-92543101
1177	Glass Injector, 4 mm ID, unpacked, single gooseneck	03-92611927

Table 3: Factory-Installed Injector Inserts

Nuts and Ferrules for Capillary Columns

Capillary columns are generally installed with a capillary column nut (P/N 03-949551-00) and a single reversed ferrule, as shown in the diagram. For connection to the TCD, a special capillary adapter (P/N 03-925605-01) is used.
A Quick Connect Kit (P/N 03-925355-90) facilitates easy connection of capillary column to Injector and detector. The kit comes with instructions and contains: column depth scale, knurled split nuts (2) and graphite jacketed ferrules (2 each of 0.4, 0.5 and 0.8 mm ID, for 250 µm, 320 µm and 530 µm columns respectively).



Three types of ferrules are common: graphite, Vespel® or polyimide and graphite/Vespel.

- Do not use graphite with ECD or GC/MS systems.
- Ferrule ID is based on the diameter of the capillary column. See table below for 1/16” ferrules.

Column ID	***Holes***	***Teflon*** ***Max 250°C***	***Vespel*** ***Max 350°C***	***40% Graphite /60% Vespel*** ***Max 400°C***	***Graphite*** ***Max 450°C***	***SilTite*** ***Metal, GC/MS***
0.18 mm ID and smaller	1	CR214103	CR212103	CR213103	-	-
0.25 mm ID	1	CR214104	CR212104	CR213104	CR211104	-
	2	-	CR212124	CR213124	CR211124	-
0.25 mm ID and smaller	1	-	-	-	-	SG073300
0.32 mm ID	1	CR214105	CR212105	CR213105	CR211105	SG073301
	2	-	CR212125	CR213125	CR211125	-
0.53 mm ID	1	CR214108	CR212108	CR213108	CR211108	SG073302

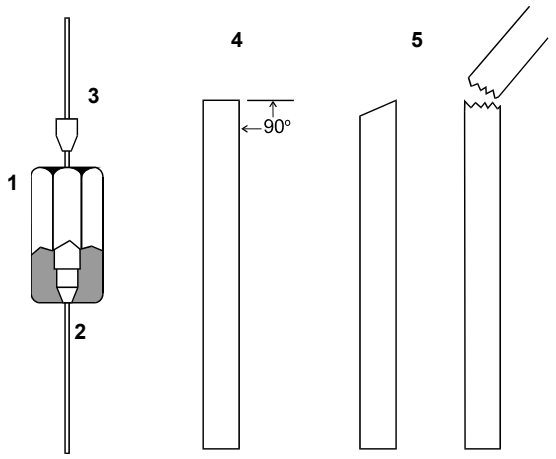
Table 4: 1/16” Column Ferrules

Connect Capillary Column to Injector

When a capillary column is used in combination with an Injector, alignment of the column is critical. Use the following steps to precisely align the capillary column. Unless this procedure is followed, the injection syringe may damage the top of the column or peak tailing may result.

To ensure a leak-free connection of capillary columns, the ends of the column must be cut squarely and as smoothly as possible. To achieve this, a proper scoring tool is required. A ceramic scoring wafer (01-900158-00) is recommended.

1. Cut off sealed section at each end of new column.
2. Uncoil 10 cm at Injector end of column.
3. Slide appropriate column nut and ferrule on each end of column. on page 14.
4. Make a clean cut with the cutting tool 1-2″ (2-4 cm) from end of the column. Inspect the cut to verify it is square and smooth.



1 Capillary column nut
2 Fused silica column
3 Capillary column ferrule
4 Properly cut column end
5 Improperly cut column ends

Figure 1: Column Ends

5. Move the nut and ferrule to within 3 cm of column end.

6. Measure the mark point (see Table 5) from the end of the column and mark this distance with a felt pen or typewriter fluid. The use of Column Scale tool (03-92575001, available in Capillary Column Quick Connect Kit on page 17, 03-925751-90) or EZ-Grip[tm] supplied with each standard GC capillary column is highly recommended.

DO NOT allow the column nut and ferrule to fall past the mark because the ink or correction fluid can contaminate the ferrule.

	1041	***1061***	***1177***	***1079 PTV***	***1093 On-Column***
Insert depth	Maximum*	Maximum*	3.7 cm	7.5 cm	8.6 cm
Mark at	None	None	2.7 cm	5 cm	5 cm

Table 5: Maximum insert depth for Capillary Column in Injector

7. * Only for 1041 and 1061Injectors, for the other Injectors continue at 12.

8. Gently push the column fully up into the Injector (9 cm) until the column comes to a firm stop. This correctly positions the column against the upper end of the Injector insert. The column must go in the full distance. If it goes not, the Injector will not function properly. Tighten the column nut just enough to prevent column movement.

9. Manually insert the syringe that will be used with the column into the 1041 Injector, checking that the syringe goes into the Injector completely with no binding or interference. If interference occurs, repeat the above column insertion procedure until the syringe moves smoothly into the Injector.

10. Continue to hold the column firmly in place as you tighten the column nut carefully and only enough to hold the column firmly in place and obtain a good seal. Remove the syringe from the Injector.

11. After the column is installed at the Injector end, make several dry injections to ensure that the needle enters the column easily.

12. Partially insert the column into the lower end of the Injector. Thread up the capillary column nut and ferrule finger-tight.

13. Gently push the column into the Injector until the mark on the column is aligned with the bottom edge of the column nut.

14. Tighten the column nut by hand until it is finger-tight. Hold the column and use a 5/16" wrench to tighten the column nut in place. Tighten the column nut only enough to seal the column and hold it firmly in place.

An optional Capillary Column Quick Connect Kit is available that simplifies the installation of a capillary column into Injectors and detectors. It contains split capillary column nuts, re-usable graphite ferrules (for 250 µm, 320 µm, 530 µm ID columns) and a column depth scale. The ferrule is set on the column using this depth scale. No felt pen or typewriter correction fluid is needed (The part number of the Quick Connect Kit is 03-925751-90).

Tips for Tightening Column Ferrules

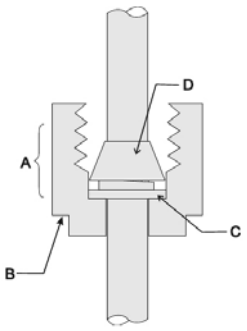
Refer to the Table 6 below for instructions to tighten the capillary column nut with a variety of ferrule types. Do not over tighten capillary column nuts use a new ferrule if the column does not seal. Over tightening capillary nuts can destroy some ferrules, particularly graphite.

Ferrule Type	*Recommendation*	*Comments*
New graphite ferrule	¼ - turn past finger-tight	¼ - to ½ -turn past finger-tight may be required to achieve a seal.
Used graphite ferrule	¼ - turn past finger-tight	If a different size of column is used, more tightening may be required.
New graphite-Vespel® ferrule	¼ - to ½ -turn past finger-tight	May require retightening after first or second programmed run. Over tightening will destroy ferrule and seal.
Used graphite-Vespel® ferrule	¼ - turn past finger-tight	Re-use only on same size column.
New Vespel® ferrule	1177/1079 PTV: ¼ - to ½ -turn past finger-tight.	May require retightening after first or second programmed run.
	Not recommended for 1041/1061/1093	
Used Vespel® ferrule	1177/1079 PTV: ¼ -turn past finger-tight.	Reuse only on same size column
	Not recommended for 1041/1061/1093	

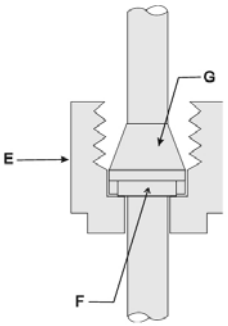
Table 6: General Tightening and Re-tightening for Common Ferrules

Nuts and Ferrules for Packed Columns

- **Metal** packed columns are usually installed with a metal 2-piece ferrule. (A commonly used metal packed column is 1/8" OD stainless steel. It uses a 1/8" ID stainless steel ferrule)
  - **A** Two Piece Ferrule
  - **B** Column Nut
  - **C** Back Ferrule
  - **D** Front Ferrule
  - **E** Column Nut
  - **F** Metal Back Ferrule Installed Backwards
  - **G** Graphite Ferrule



- **Glass** packed columns are 1/4" OD and are installed with a graphite front ferrule and a metal back ferrule installed backwards.



Connect a Packed Column to the Injector

Follow the steps below to install a new packed column into your 1041 or 1061 Injector. Note that the Injector must first be converted for packed column use by installing the appropriate adapters.

- Place nuts and ferrules on both ends of column as shown.
- If your column is metal, insert it into the Injector fitting and gently push until it stops. Manipulate the column so the detector end points to the detector
  OR
- If your column is glass, insert it into the Injector, rotate it slightly so the detector end is out of alignment with the detector and push gently until the end is near but not touching the oven ceiling.
- Hold the column in place and thread the nuts by hand.
- Tighten the nut finger-tight and then give it a further 3/4-turn with a small wrench.

Varian Test Column

At points throughout this manual, specific values and conditions are given for the analysis of Varian Test Samples on Varian Test Columns. Table 7 provides information on Varian Test Columns.

Column Description	*For use with Injector Models*	*Part Number*
10% SE30 WHP 80/100 1mx1/8" UM 1041 PREC	1041/1061 Packed Adapters	-
Capillary 15M x 0.25id VF-1ms, df=0.25um	1177 or 1079 PTV	CP8907
VF-1ms FS 15 x .25mm Df=0.25µm	1177 or 1079 PTV	CP8907 (ECD)
Capillary 10M x 530 id CP-Sil 5 CB fused silica	1041/1061	CP7620

Table 7: Varian Test Columns

Identify Carrier Gas Controls and Turn on Flow

Gases are plumbed at the factory through the rear bulkhead fittings into the inlet manifold, then to the pneumatic controls in the pneumatics compartment. The pneumatic controls can be either electronically controlled (EFC) or manually controlled and are different for each Injector system.

If your Injector model is a ...	*Carrier gas flow is regulated by a ...*
1041/1061	Type EFC23 or type EFC24 with valving or Flow controller (controls flow into Injector) *and a* Back pressure regulator (controls flow through column)
1177/1079 PTV/1093	Type EFC21/EFC25 **or** Flow controller (controls flow into Injector) *and a* Back pressure regulator (controls flow through column)

Table 8: Injector and EFC type combination

- Carrier gas flow is adjusted at the user interface for EFC or at the pneumatics panel for manual pneumatics. For manual pneumatics carrier gas systems, identify the controls for carrier gas flow at the pneumatics panel for your Injector system. These pneumatic controls are identified by their location, i.e. front, middle, rear.
- Turn on carrier gas flow by adjusting the manual flow controller or setting a positive pressure or flow on the EFC system.
- Verify that there is a positive pressure on the pneumatics panel pressure gauge or FLOW/PRESSURE page Press .

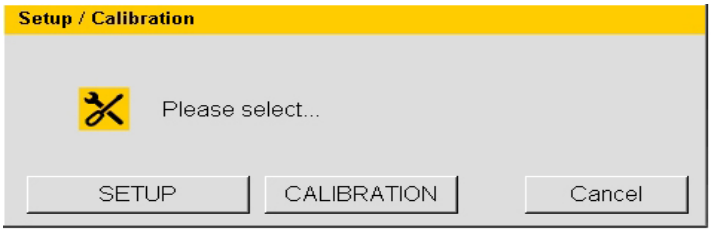
Split Vent Flow and Septum Purge Flow

Whether you are making an injection in the split or splitless mode, a split vent flow rate needs to be set. In this example the column flow is set to 1 mL/min, the split vent flow is set to 100 mL/min corresponding to a split ratio of 100. A split ratio of 100 means that for every mL of gas through the column, 100 mL/s will flow out the vent. The split state is set in the Injector section of the 450-GC method in case an EFC is installed or in the VALVE TABLE section of SAMPLE DELIVERY in case an manual pneumatics is installed. In addition to a split state a septum purge flow rate should also be set. The following information is for setting a split vent flow rate with EFC installed. For either a 1177 or 1079 PTV with manual pneumatics see the Injector section of the Operator's Manual on page 97 for further information.

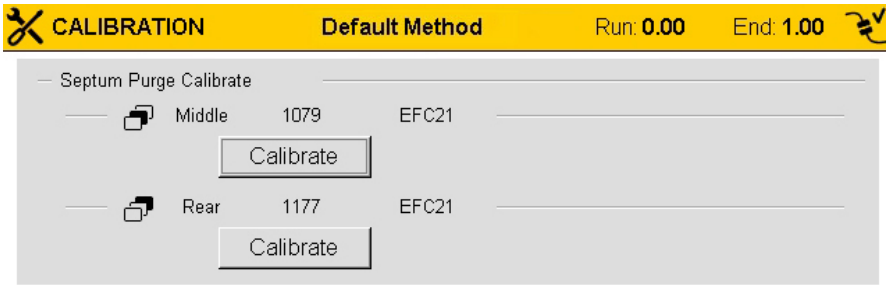
1. Press the FLOW / PRESSURE key and set the appropriate head pressure for your analytical column.
2. Press the INJECTOR key and select either the 1177 or the 1079 PTV Injector by location.
3. When EFC is installed, in addition to programming the split state, the split ratio can also be programmed.
4. Set the initial split state to ON and set the split ratio to 100.
5. Press the COLUMN OVEN key. Turn the column oven off by pushing on the button "Turn Oven Power Off".
6. Allow the oven to cool down. Open the column oven door and adjust the septum purge needle valve located above the column oven until the flow rate is in the range 3 - 5 mL/min. For EFC equipped systems this value has to be entered in the septum purge calibration routine.

Septum purge calibration

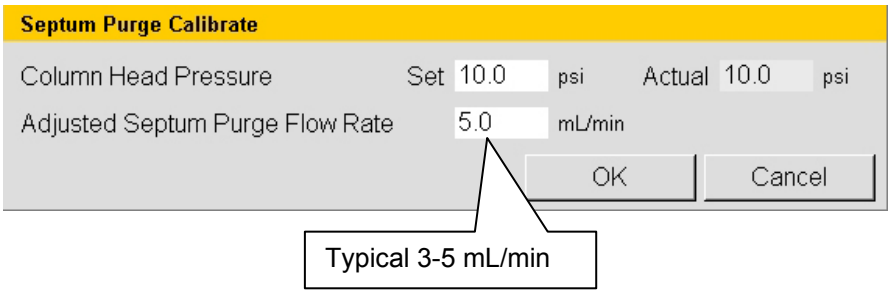
- Select the SETUP key and select CALIBRATION.



- Select the Injector you are setting up by specifying the location (front, middle or rear) or type.



- Press the “Calibrate” button.



- You will be prompted to enter the Column Head Pressure and Adjusted Septum Purge Flow Rate. Fill in the column head pressure and adjust and measure the Septum Purge Flow rate.
- Fill in the actual Septum Purge Flow and press the “OK” button.

Set Carrier Gas Flow Rate to Condition Column

To condition the column, flow rates need only be approximate. For packed columns and wide bore (0.53 mm ID) capillary columns set the flow rate to 10 - 20 mL/min. Press the FLOW / PRESSURE key, select the appropriate Injector by location and set the initial flow to 10 - 20 mL/min (15 mL/min for a wide bore column and 20 mL/min for a packed column).
Use the following guide to set an appropriate column flow for either the 1177 or the 1079 PTV Injector with narrow bore capillary columns. With manual pneumatics the pressure is set using either the 1177 or the 1079 PTV back pressure regulator on the pneumatics panel. If the Capillary Injector has EFC press the FLOW / PRESSURE key, select the appropriate Injector by location and set the pressure according the table:

Column ID	*Head Pressure*
250 µm	170 kPa (25 psi)
320 µm	100 kPa (15 psi)
530 µm	35 kPa (5 psi)

Table 9: Column condition head pressure

For capillary columns, the gasflow through the column is verified by dipping the detector end of column into a beaker containing a solvent, such as isopropanol. Positive carrier gas flow should be indicated by a stream of bubbles.

Injector Conditioning

It may be necessary to bake out the Injector if it has become contaminated during the re-assembly process, i.e. if the internal parts were placed on dirty surfaces or touched with bare hands. Then, both the Injector and column should be conditioned. See section Maintenance, Instructions for High-Temperature bakeout of Injectors on page 433.

To prevent contamination and overheating of the fused silica column, the column must NOT be connected to the Injector during the Injector bake-out procedure.

Column Conditioning

In general the column should be conditioned at 20°C above the maximum temperature used in the analytical method, or 20°C below the maximum operating temperature of the column, whichever is lower. Check with the column supplier for the correct conditioned details.

New Columns

To minimize chemical contamination of the detector, all new columns should be baked out before they are connected to the detector. Refer to the supplier's recommendations for temperatures.

Used Columns

Periodically (especially during extended isothermal operation), the column should be baked out to remove accumulated contaminants. Column conditioning is required when column efficiency begins to fall off, i.e. loss of resolution, peak tailing, severely drifting baseline, retention of extra peaks, etc. For fused silica columns, it is recommended that 1 to 2 turns at the beginning of the column (approximately 1/2 meter) be cut off prior to reconditioning, as this is the end part of the column where the stationary phase has most likely been disturbed. Attach the column to the Injector and, with carrier gas flowing, bake out the column for a minimum of 3 hours.

It is important to establish the maximum recommended conditioning temperature for the installed column. This information is provided with the column or can be obtained from the column manufacturer. The maximum allowable temperature for the column does not have to be used for conditioning purposes.

A GC column may be irreversibly damaged if it is heated with air or moisture present in the pneumatic system. It is important to purge the column with inert carrier gas before heating the column. After the column flow is established, allow the carrier gas to purge through the column for 15 - 20 minutes before heating it.

Connect Capillary Column to Detector

After the column has been conditioned, follow the steps below to connect the capillary column to the detector.

WARNING:
BURN HAZARD

Hot surfaces are exposed

- Check that the capillary column nut and ferrule are installed on the column. Note that if the TCD is used with capillary columns, a special capillary adapter (PN 03-925605-01) is used on the TCD inlet.

DO NOT use graphite ferrules with the ECD.

- Uncoil about 20 cm from end of column.
- Move the column nut and ferrule to within 5 cm of the column end. Find the column insertion depth for your detector in Figure below. Measure the correct distance from the end of the column and mark this distance. Refer to diagram below for correct distances. *DO NOT let the column nut and ferrule fall past this mark. The marking medium can contaminate the ferrule.*

Column Insertion Depths for Varian Detectors

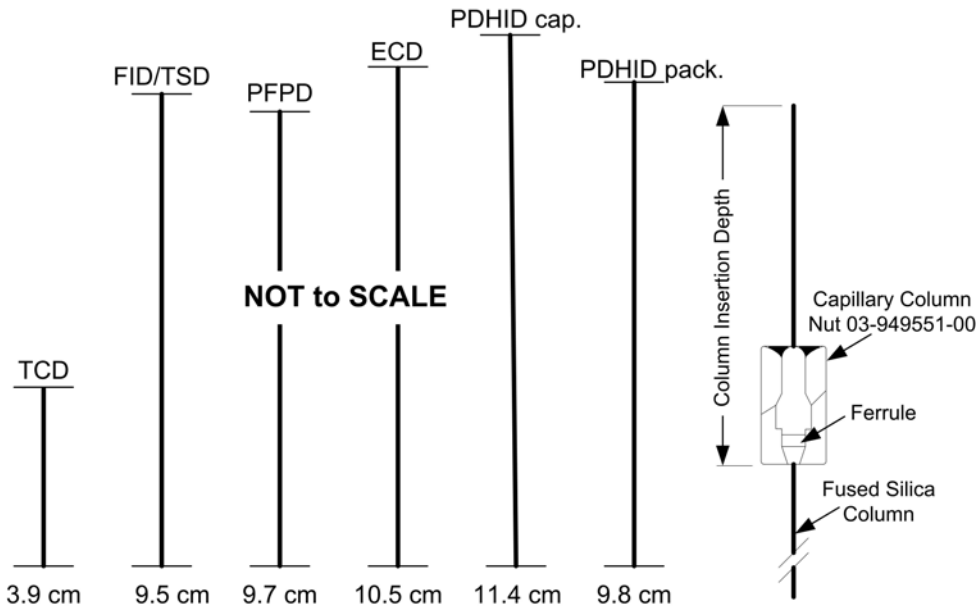


Figure 2: Column Insertion Depths for Detectors

- Place the column into the base of the detector.
- Turn the capillary column nut and ferrule finger-tight.
- Gently slide the column into the detector until the mark on the column aligns with the bottom edge of the column nut.
- Continue to hold the column as you tighten the nut just enough to hold the column firmly in place and to create a good seal.

Connect Metal Packed Column to Detector

After the column has been conditioned, follow the steps below to connect your metal packed column to the detector. Note that the appropriate packed column adapter must be installed.

WARNING:
BURN HAZARD

Hot surfaces are exposed

- If using a detector other than TCD, locate the packed column detector adapter in the Packed Column Kit and install on the inlet to the detector. The TCD inlet is designed for packed column installation.
- Insert the open end of the packed column into the detector inlet or adapter and push until it reaches a stop.
- Hold the column in place and finger-tighten the column nut. Then, using 2 7/16″ wrenches, tighten 3/4-turn past finger-tight. On subsequent installations, tighten the nut 1/4 to 1/2-turn past finger-tight.

Check for Leaks

Inspect your GC system for leaks before heating. An electronic leak detector is the most reliable method to check the Injector and detector fittings for carrier gas leaks.

CAUTION

Never use soaps, such as Snoop®. These may contaminate the system and cause permanent damage to capillary columns. If a liquid leak detector is desired, try a 50/50 mixture of isopropanol/water.

BASIC OPERATION

At points throughout this section, specific values and conditions are given for analysis of the Varian test samples on Varian test columns. Your analytical column and application may require different values and conditions. Please ascertain the range of operating temperatures for your analytical column and make sure your settings are within this range.

Setting Carrier and Detector Gas Flow Rates

The following table indicates the typical values for carrier and detector gas flow rates. For practical purposes a generalized carrier gas flow rate of 2 mL/min is used for capillary columns. Note that the carrier gas and make-up combined flow is typically 30 mL/min.

Detector	*Carrier Gas*	*Carrier Gas Flow Rate (mL/min)*		*Make-Up Gas*	*Make-Up + Carrier Gas Flow Rate (mL/min)*	*Detector Gas Air 1 (mL/min)*	*Detector Gas Air 2 (mL/min)*	*Detector Gas H_2 (mL/min)*
		Packed	*Capillary*					
FID	N_2, H_2 or He	20 ±1	1 - 15	N_2 or He	30 ±1	300 ±10	—	30 ±1
PDHID	For more details see Installation Manual PDHID							
TSD	N_2 or He	20 ± 1	1 - 15	Same as Carrier Gas	30 ±1	175 ±10	—	4 ±0.2
TCD[a]	N_2, He or H_2	20 ±1	1 - 15	Same as Carrier Gas	30 ±1	—	—	—
ECD	N_2 or Ar/CH_4	20 ±1	1 - 15	N_2 or Ar/CH_4	30 ±1	—	—	—
PFPD	N_2, He or H_2	20 ±1[b]	1 - 10	None	—	17 ±1	10 ± 1	13 ±1

Table 10: Flow rates for Carrier and Detector Gases

[a] **TCD Reference Flow** – The flow rate for carrier gas through the reference cell is 30 ±1 mL/min.

[b] **PFPD with packed columns** – An effluent splitter is used to reduce the carrier flow to the detector to < 5 mL/min.

Follow the steps below to set all gas flow rates on the 450-GC.
Flows are typically set with carrier gas first followed by detector make-up and fuel gases. The most convenient way to measure or verify gas flows is at the detector exit.
Adapters are provided in the individual detector accessory kits for this purpose. Note, however, that the most accurate method to determine FID and TSD flows is at the flame tip after removing the tower.

- Attach a bubble or digital flowmeter to the detector outlet using the appropriate adapter.
- Turn off make-up and detector gases at the pneumatics panel. For a needle valve this is accomplished by turning the needle valve cartridge knob fully clockwise. For a flow controller this is accomplished by turning the flow controller knob fully counter-clockwise or until the turns counting dial reads zero.
- Observe reading on flowmeter. Ensure it is within the range described in the table for carrier gas flow rates.
- Adjust the carrier gas flow using the manual pneumatics controls on the pneumatics panel or by adjusting the Electronic Flow Control to achieve the desired flow rate.
- For a capillary system, turn on the make-up needle valve and adjust to achieve the desired flow rate. The combined carrier and make-up flow should be 30 mL/min. Adjustment is done with a small flat screwdriver inside the needle valve cartridge knob.
- Turn on the relevant detector fuel gases in the sequence hydrogen followed by air. Use the table below to calculate the combined flows for the specific detector. For the TSD adjust the hydrogen flow controller until the turns dial reads 400.

Detector	***Carrier + make-up***	***Carrier + make-up + hydrogen***	***Carrier + make-up + hydrogen + air1***	***Carrier + make-up + hydrogen +air1 + air2***
FID	30	60	360	
TSD	30	34	209	
PFPD	2	15	32	42

Table 11: Detector manual Flow rates

Condition the GC System

As the Injector and column were conditioned earlier, this procedure represents a quick conditioning of the entire GC system including the detector. Follow the steps below to build an analytical method to condition the GC system.

- Press the INJECTOR key and verify that the Injector temperature is 250°C. If the Injector is either a 1177 or a 1079 PTV verify that the initial split state is ON and that there is no split time program. Set the split ratio to 100.
- Press the COLUMN OVEN key and set the initial column oven temperature to 250°C.
- Press the DETECTOR key and set the detector oven temperature to 300°C. If the detector is an FID, wait for the detector zone to get above 100°C (check here on page 287 for more Ignite conditions) and then press the ignite button to ignite the flame.
- Allow the system to condition for one hour.

Build a Method to Analyze a Test Sample

The conditions for running a test sample depend on the sample chosen. The test sample is typically one of the following:

- The column test mixture supplied by the column manufacturer,
- a Varian Test Sample, or
- a test mixture you select as an indicator of system performance for a particular analysis.

Method to Analyze a Varian Test Sample on a Varian Test Column

Test Sample	*Part Number*	*Concentrations of Test Compounds*
TCD	82-005048-01	3.00 μg/μL of C_{14}, C_{15} and C_{16} in iso-octane.
ECD	82-005048-02	33.0 pg/μL of lindane and aldrin in iso-octane.
PFPD	82-005048-03	20.0 ng/μL of n-dodecanethiol, tributylphosphate, methyl parathion; 4000 ng/μL of n-pentadecane in iso-octane.
TSD	82-005048-04	2.00 ng/μL of azobenzene, methyl parathion; 4.00 ng/μL malathion and 4.00 μg/μL C_{17} in iso-octane.
FID (Capillary Column)	82-005048-07	30 ng/μL of C_{14}, C_{15} and C_{16} in iso-octane.
FID (Packed Column)	82-005048-00	3 ng/μL of C_{14}, C_{15} and C_{16} in iso-octane.
Note: *If the FID test sample is not available, the TCD test sample can be used if first diluted 100:1.*		

Table 12: Varian Test Samples

Note: *These conditions may not conform to the requirements of your analytical column. Do not exceed your column's T_{max}. See Column Manufacturer's specifications for your column.*

For 1041 and 1061 Injectors

	ECD	*FID*	*TCD*[a]	*TSD*	*PFPD*
Initial column oven temp (°C)	80	80	80	80	80
Initial column oven hold time (min)	1	1	1	1	1
Program 1 ramp rate (°C/min)	20	20	20	20	20
Final column oven temp (°C)	200	200	200	200	200
Final column hold time (min)	5	5	5	5	5
Initial column oven temp (°C) (Isothermal operation)	190	140	140	175	175
Initial column oven hold time (min) (Isothermal operation)	15	15	15	15	15
Injector temp (°C)	220	220	220	220	220
Detector temp (°C)	300	300	220	300	300[b]
Range	1	12	0.05	12	10[c]
Initial Autozero	On	On	On	On	On
TSD bead current (amps)				3.0 ±0.3	

Table 13: Varian Test Sample 1041 and 1061 Conditions

[a] TCD Reference Flow – Rate for carrier gas flow through the reference cell is 20 ±1 mL/min.
[b] Depends on application: S-mode 200°C, P-mode 300°C and N-mode 250°C.
[c] Normal range: 10 if Varian testmix is being used.

For 1177 and 1079 PTV Injectors

	ECD	*FID*	*TCD*[a]	*TSD*	*PFPD*
Initial column oven temp (°C)	80	80	80	80	80
Initial column oven hold time (min)	1	1	1	1	1
Program 1 column oven final temp (°C)	200	200	200	200	200
Program 1 column oven ramp rate (°C/min)	20	20	20	20	20
Program 1 column oven hold time (min)	5	5	5	5	5
Injector temp (°C)	220	220	220	220	220
Detector temp (°C)	300	300	220	300	300[b]
Range	1	12	0.05	12	10
Initial Autozero	On	On	On	On	On
TSD bead current (amps)	-	-	-	3.0 ±0.3	-
Injection mode	Splitless				
Initial split state	ON				
Step 1 time (min)	0.01				
Step 1 split state	OFF				
Step 2 time (min)	1.00				
Step 2 split state	ON				
Step 2 split ratio	20				

Table 14: Varian Test Sample 1177 and 1079 Conditions

Prepare the Test Sample

You are now ready to prepare your sample. For preparation of samples for automated injection, please refer to the CP-8400 AutoSampler section on page 121.

You may inject your sample neat or diluted in a solvent. The Varian PFPD test sample should be diluted 1:30 in iso-octane if you intend to use it for a splitless injection. The choice of solvents depends on a number of factors, including component solubility, type of detector and polarity of the analytical column. Select solvents that match the polarity of your column.

[a] TCD Reference Flow – Rate for carrier gas flow through the reference cell is 20 ±1 mL/min.
[b] Depends on application: S-mode 200°C, P-mode 300°C and N-mode 250°C.

Solvents for GC

Following are some recommended solvents for non-polar, intermediate and polar phase columns.

Column Phase	*Recommended Solvent*	*Boiling Point (°C)*
Non-Polar	Pentane	36.1
100% Methyl	n-Hexane	69.0
5% Phenyl, 95% Methyl	Cyclohexane	80.7
	Iso-octane	99.3
	Benzene	80.1
	Toluene	110.6
	Ethyl Ether	34.6
	Methyl tert-butyl ether	55.2
	Methylene Chloride	39.8
	Chloroform	61.2
	Carbon Disulfide	46.5
Intermediate	Ethyl Acetate	77.0
50% Phenyl, 50% Methyl	Acetone	56.5
	Methyl iso-butyl ketone	127.0
	Acetonitrile	81.6
Polar	Methanol	64.7
Polyethyene Glycol	Ethanol	78.5
	2-Propanol	82.5
	n-Butanol	117.7

Table 15: Recommended Solvents for Gas Chromatography

Sample Volume and Injection Rate

The quality of your chromatogram can be greatly affected by the sample volume and injection rate. These vary according to the type of Injector and column you are using.
See also in section: Technical Tips, Choosing the right GC Injection technique, on page 466. Shown below are some recommended sample volumes and injection rates. Draw the appropriate volume of sample into your syringe and inject at specified rate.

Injector	**On-Column, Flash Vaporizing (1041/1061)**	**Split (1079 PTV, 1177)**	**Splitless (1079 PTV, 1177)**
Column	Wide bore Capillary Columns (0.53 mm ID) Packed Columns	All Capillary Columns	All Capillary Columns
Sample Volume in µL	0.1–2 Wide bore Capillary 1-5 Packed Columns	0.2-2	0.2-1
Injection Rate, in µL/sec	0.5-5 Wide bore Capillary Column	25-50 for CP-8400/8410 A/S	1 for CP-8400/8410 A/S

Inject the Test Sample

Use 1µL of the Varian test sample for all detectors. In addition, if you are using a PFPD, make a 1:20 dilution of the test sample in iso-octane. With the READY light on at the GC and the appropriate volume of test sample in your injection syringe, you are ready to make your injection. For automated injection of samples, please refer to the CP-8400 AutoSampler section.

- To inject your sample, insert the syringe needle into the Injector inlet, press the syringe barrel against the Injector switch and depress it completely, simultaneously pressing down the syringe plunger. Remove the syringe from the Injector immediately.
- The blue RUN light starts to blink until the analysis ends. The run may be stopped by pressing the STOP key.
- Compare the resulting chromatogram with your known test chromatogram.
- If you are analyzing the Varian Test Sample on a Varian Test Column, you may compare your resulting chromatogram with the appropriate test chromatogram in the Detector Test Chromatograms, on page 419. The retention times will be close to those shown in the example chromatogram. The peak heights may be considerably different.
- Failure to duplicate the results you expected may indicate an installation, operation or instrument problem. This problem must be corrected before proceeding.

REFERENCE

PERIPHERAL CONNECTORS

The 450-GC Gas Chromatograph is equipped with various connectors on the rear panel and top of the instrument intended for use with peripheral equipment such as AutoSamplers and data handling devices. The following are the standard connectors on the 450-GC and their intended uses:

PERIPHERAL CONNECTORS

Top View

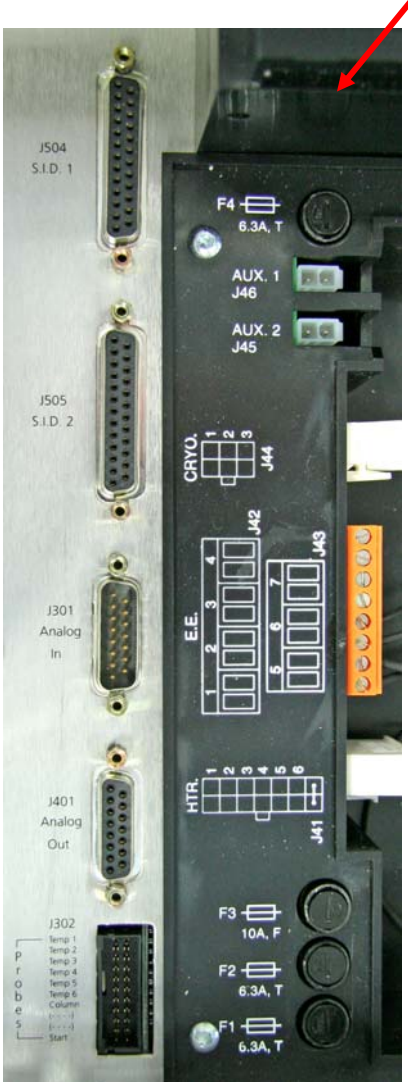
Peripheral connectors are located underneath the detector cover. Lift the detector cover up at the on/off power-switch and slide the cover to the rear of the GC. Lift the cover up and remove it.

J504/J505 S.I.D. 1 and 2
Sample Introduction Device, used for autosampler control, See on page 35.

J301 Analog IN
Not in use

J401 Analog OUT
Analog signals for Integrators (option)
See on page 36

J302 Temperature Probes
Thermocouples for the Heaters
See on page 37



Fuse F4, 6.3A, T see on page 44

J45/J46 Auxiliary 1 and 2
24 VAC output, see on page 38

J44 Cryo Valves (3)
24 VDC Cryo valve connection, See on page 39

J42/J43 External Events
Timed Events contacts
See on page 40

J41 Heater Connections
Line-Voltage Heater connections
See on page 41

Fuse F3, 10A, F see on page 44

Fuse F2, 6.3A, T see on page 44

Fuse F1, 6.3A, T see on page 44

Figure 3: Connectors for Peripheral Equipment

J504/J505 S.I.D. 1 AND 2

Sample Introduction Device, used for AutoSampler control.
AutoSampler CP-84XX **ONLY** on connector S.I.D. 2, CombiPal on connector S.I.D. 1 or S.I.D. 2.

Top View

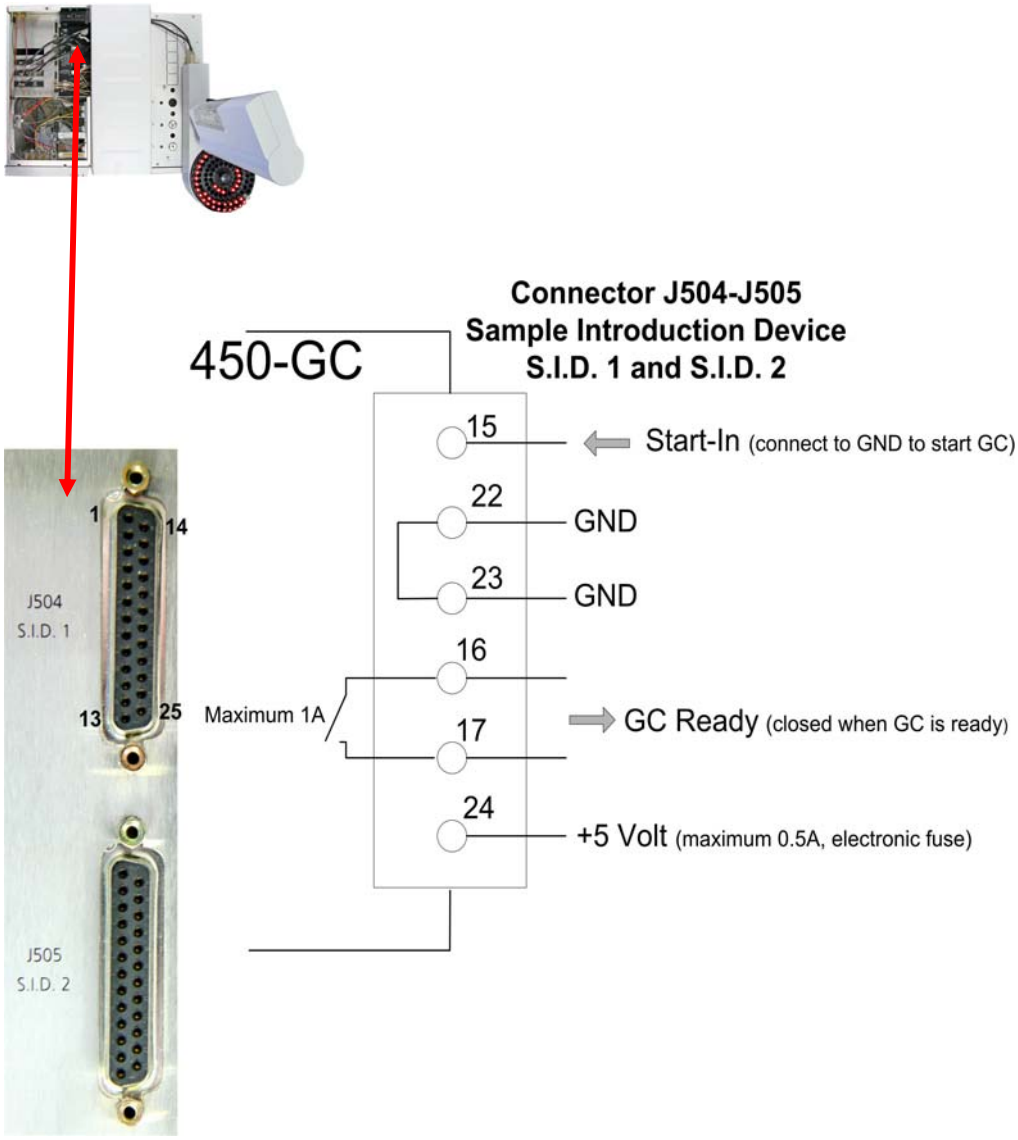


Figure 4: Connector J504-J505

J401 ANALOG OUT

Analog signals for Integrators

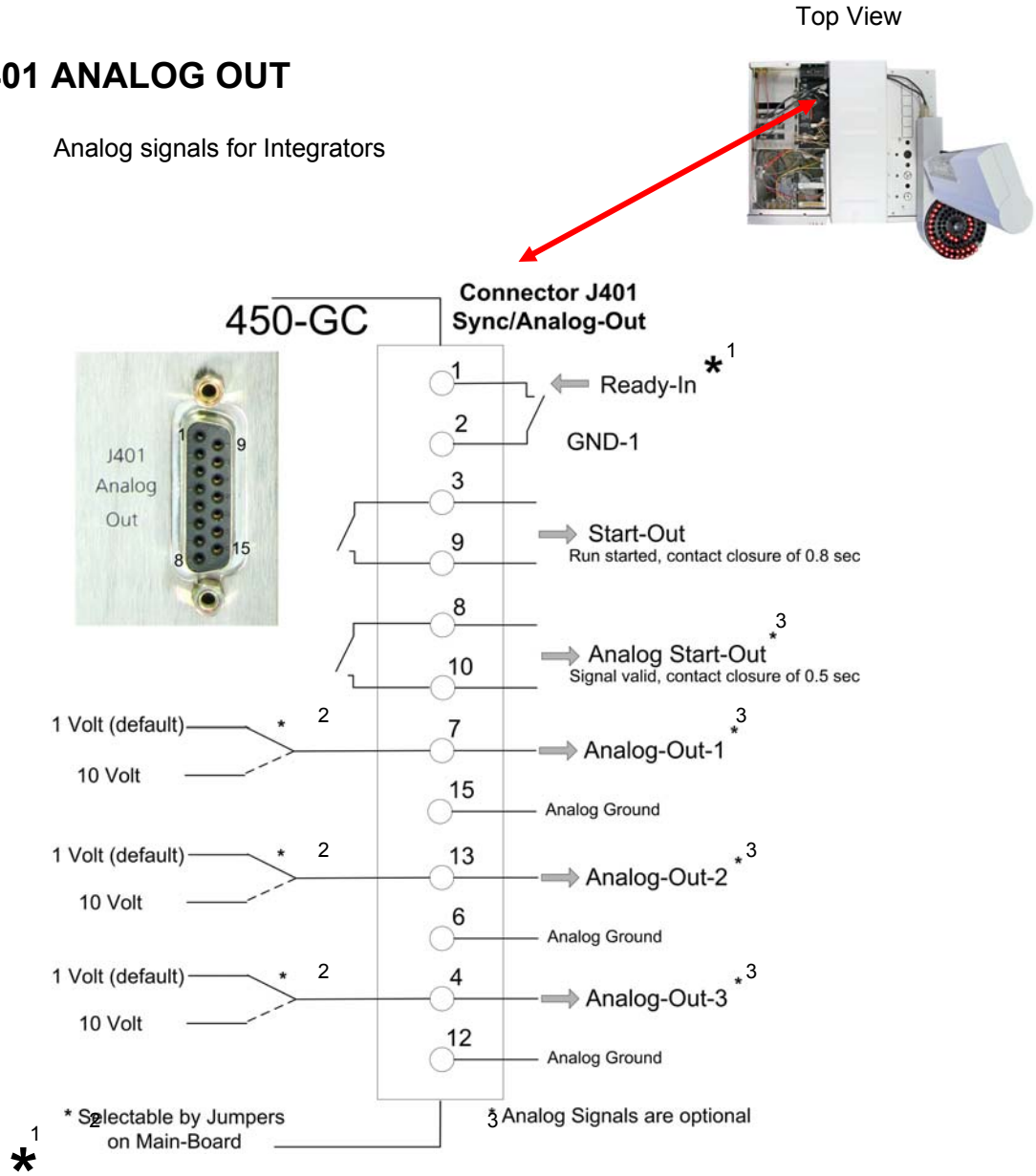


Figure 5: Connector J401

If an Integrator or other device is connected, the 450-GC is waiting (not ready on display) for Ready-In signal (pin-1) to start.
In the Setup tab Configuration, on page 67 the Ready-In Closed Contact checkbox must be checked! Available Analog cables on page 94.
0 – 1 Volt default range used for all detectors except the FID (0 – 10 Volt).

J302 TEMPERATURE PROBES

Temperature probe connectors for Heaters

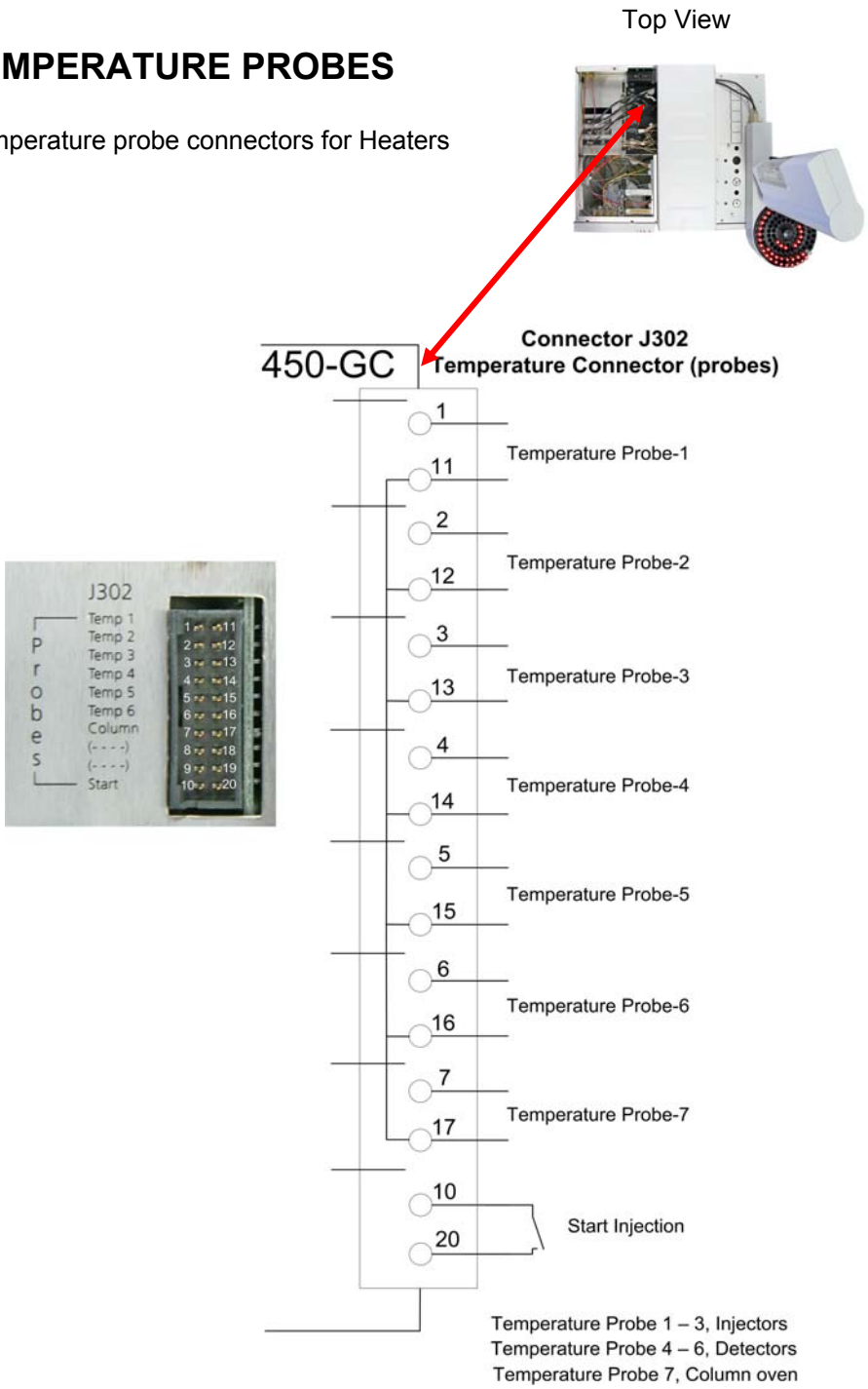


Figure 6: Connector J302

J45/J46 AUXILIARY 1 AND 2

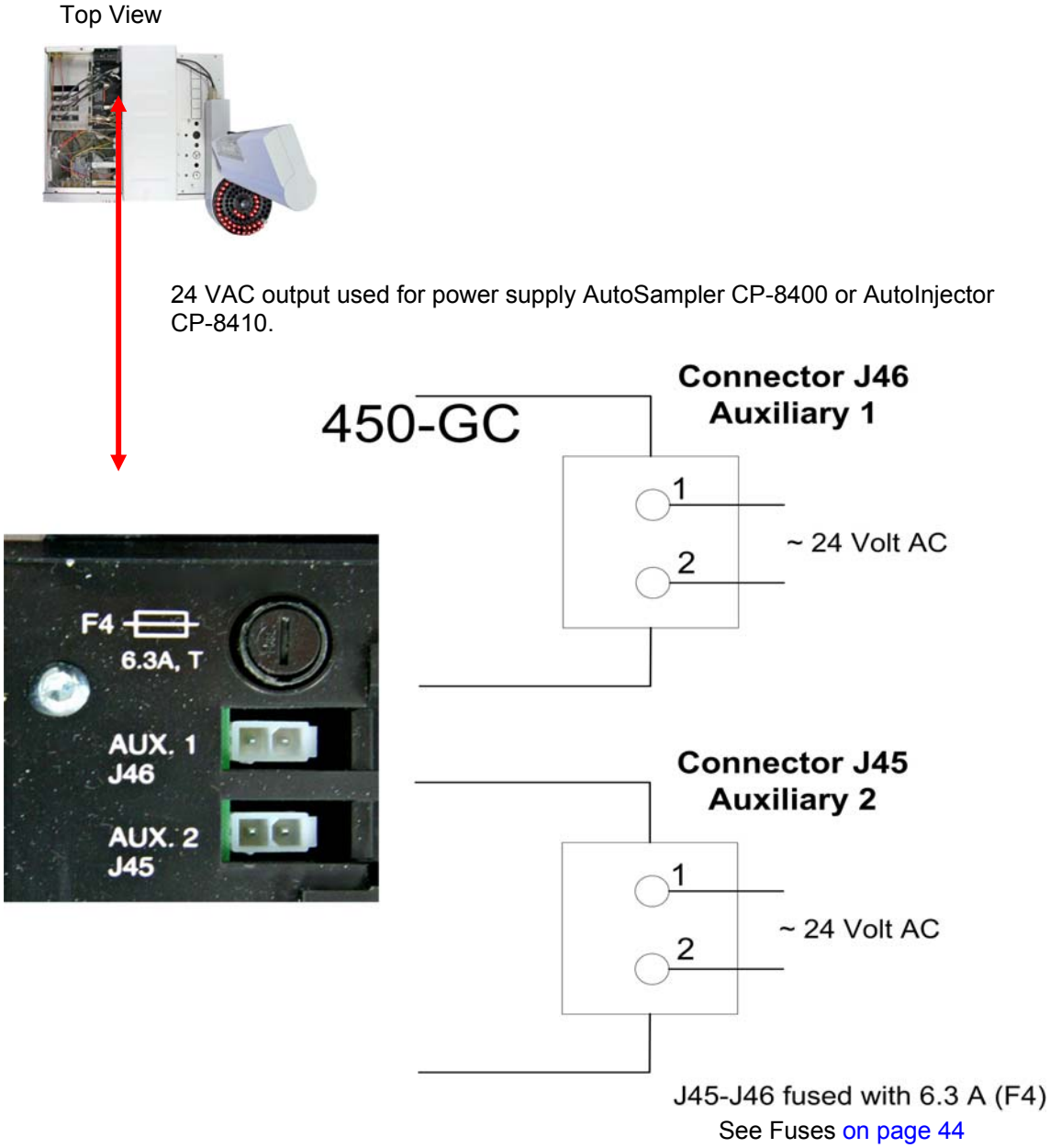


Figure 7: Connector J45/J46

J44 CRYO VALVES (3)

24 VDC Cryo valve connections

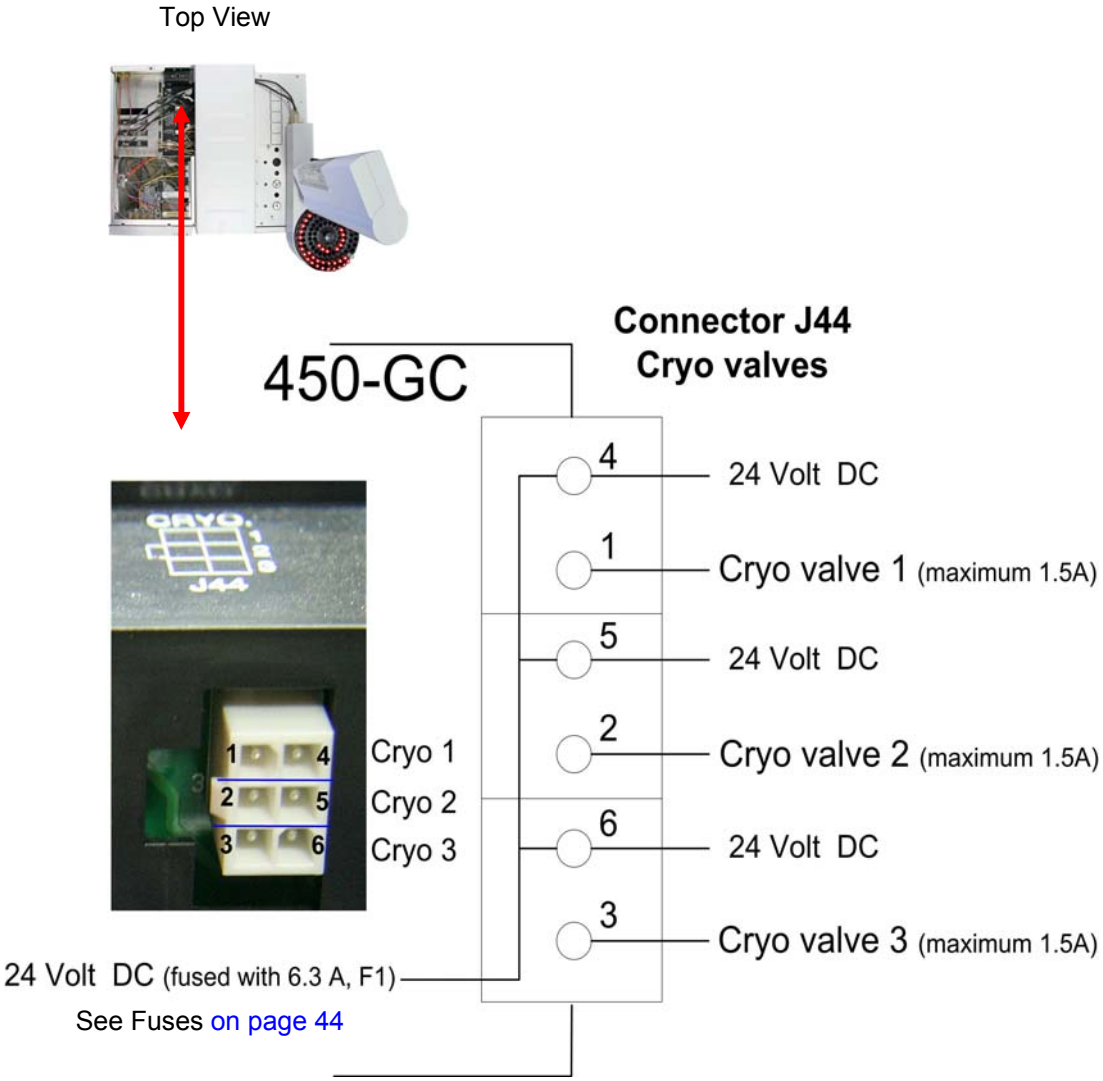
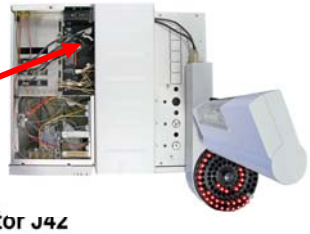
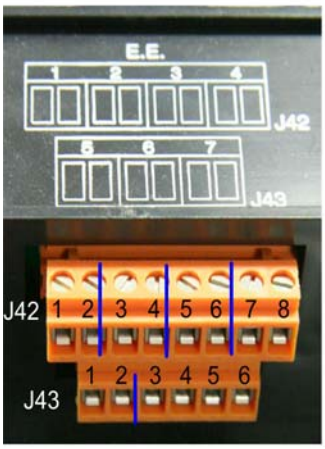


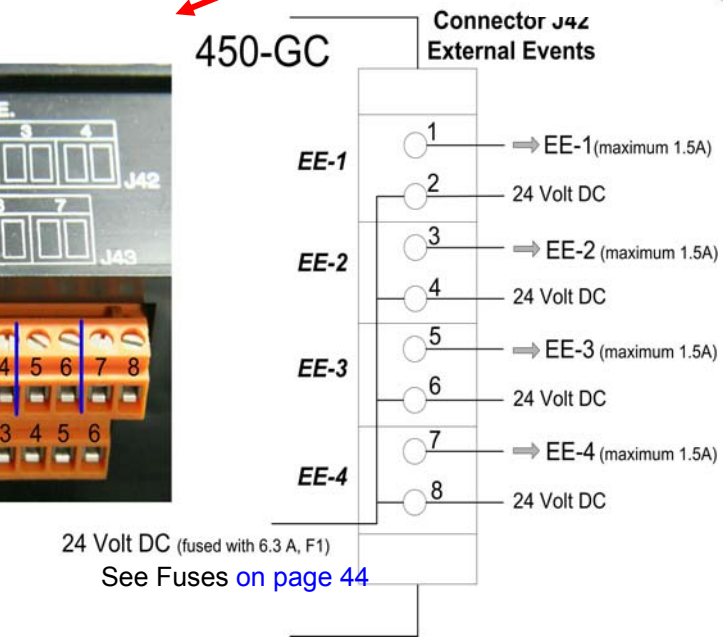
Figure 8: Connector J44

J42/J43 EXTERNAL EVENTS

Timed 24 Volt (DC) contacts (7) connector, for more (optional) contacts see Option Board on page 43.







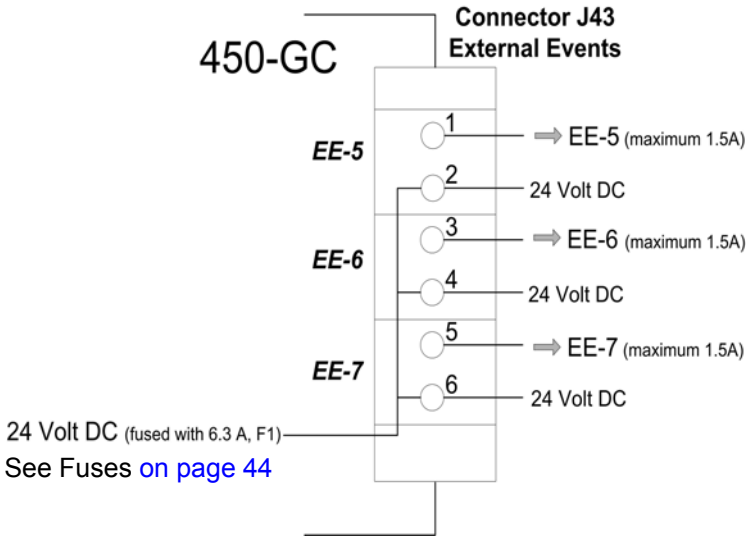
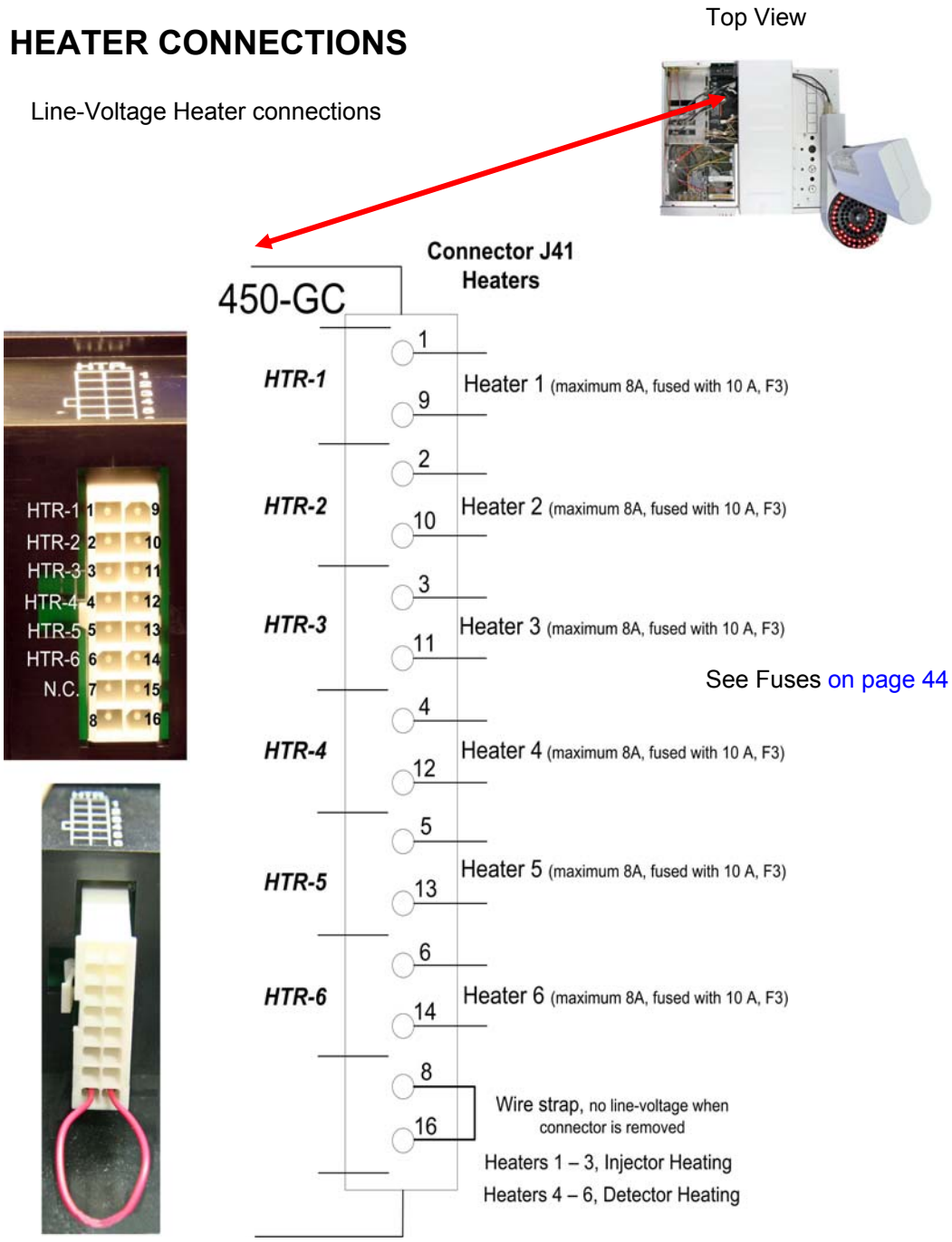


Figure 9: Connector J42/J43

J41 HEATER CONNECTIONS

Line-Voltage Heater connections

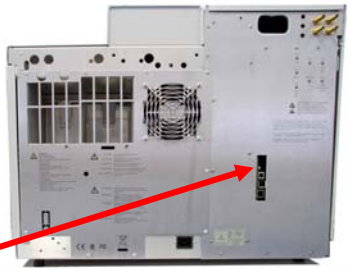


See Fuses on page 44

Figure 10: Connector J41

Rear View

REAR PANEL



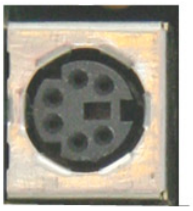
DHCP

Switch between “DHCP” and “Fixed IP”.
The **Dynamic Host Configuration Protocol** (**DHCP**) is a set of rules used by a communications device such as a 450-GC to allow the device to request and obtain an IP address from a server which has a list of addresses available for assignment.

Fixed IP

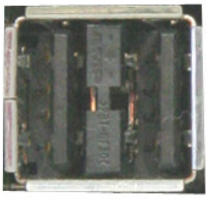
Fixed IP, (Internet Protocol address) is a unique fixed address that the 450-GC uses in order to identify and communicate with a computer network.

See Setup, tab “System” for more details.
Contact the network administrator for more information.



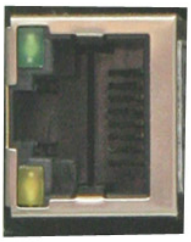
Keyboard

Not in use.



USB

Not in use.



Ethernet

Ethernet RJ45-style connector, used to connect the 450-GC to a computer, network or other devices.
Green led: cable correct connected
Yellow led: Transmit/Receive data.

OPTION BOARD

The Option Board is situated below the top-cover in the Option Slot.
The Option Board have 8 Time-Events contacts (24 Volt DC) available, also 3 Analog Outputs are present and available via connector J401 Analog Out.

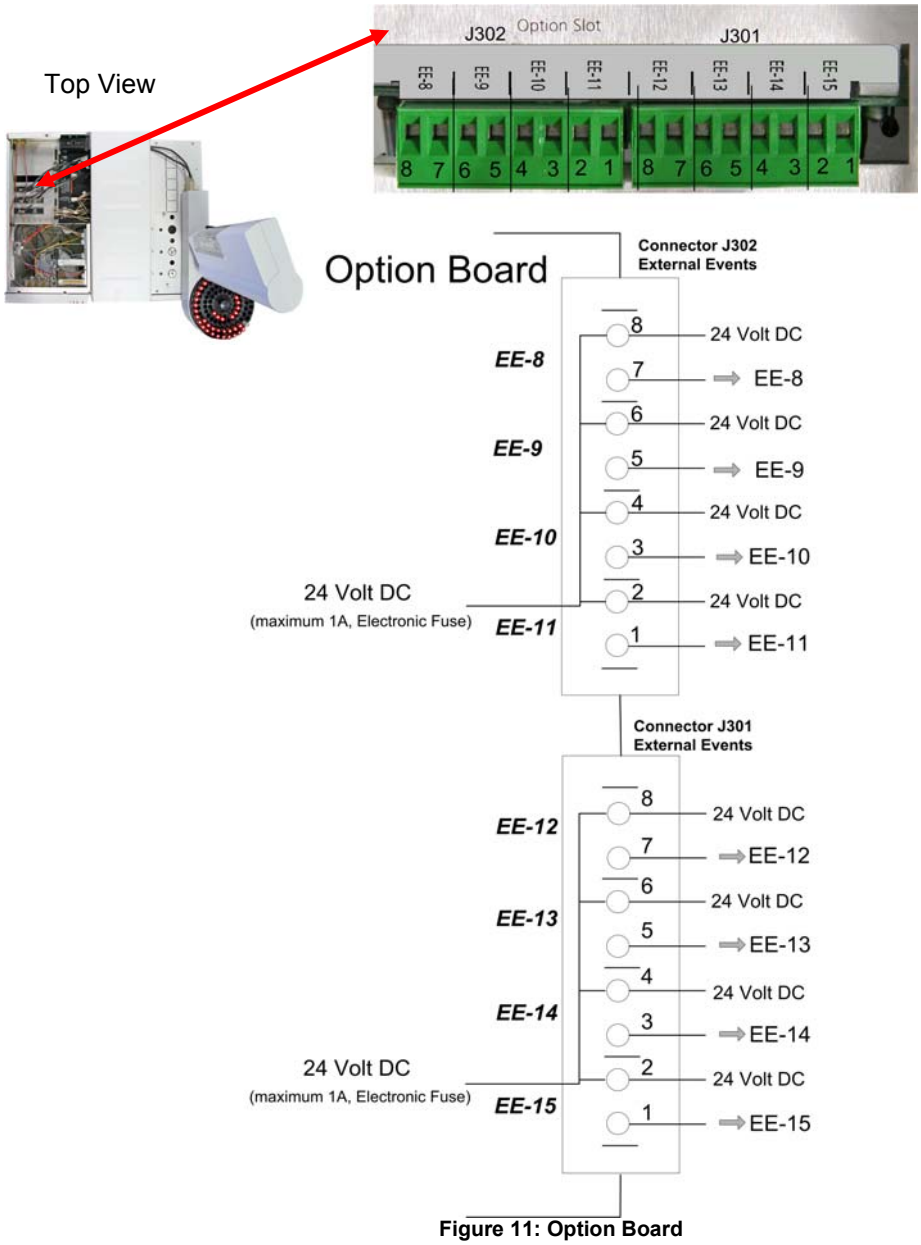


Figure 11: Option Board

FUSES COMMON

Fuses have been installed within the instrument for the protection of major power circuits.
They are designed to open as quickly as possible to prevent damage to other components within the circuit.
Occasionally, an opened fuse may have been caused by a short one-time surge; however, it is far more common that a component within the protected circuit has failed.

- When an open fuse is noticed, replace it.
- If the replacement blows, suspect a component failure.

WARNING

Before replacing fuses, turn off the oven and any heated zones and let them cool down. Turn off any detector gases at their supply. Then turn off the main power switch and unplug the power cord.

Fuses

The major fuses are located on the power module.
There are 4 fuses on the power module on page 34
Fuse F5 is an electronic fuse and situated on the electronic main board (not accessible or replaceable).

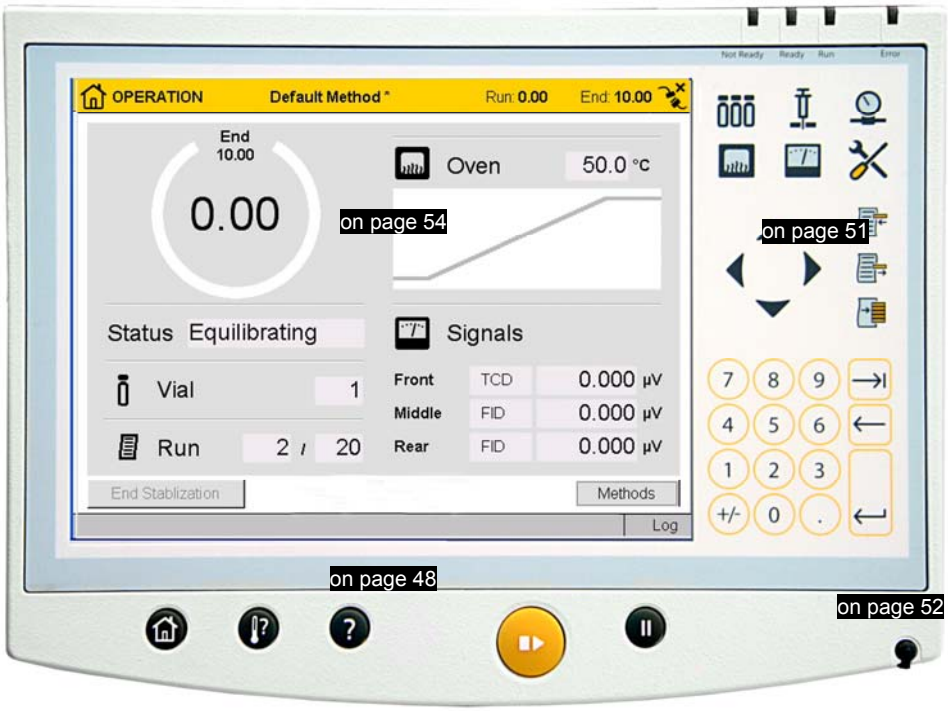
ID	*Description*	*System*	*Power Rating*	*Partnumber*
F1	Glass/Type T	24VDC External Events	6.3A/250V 5 x 20 mm	5550033400
F2	Glass/Type T	24VDC External Events	6.3A/250V 5 x 20 mm	5550033400
F3	Glass/Type F	Heaters/Ovenfan (except column oven)	10A/250V 5 x 20 mm	5550034800
F4	Glass/Type T	24 VAC Auxiliary connectors (Sampler)	6.3A/250V 5 x 20 mm	5550033400
F5	Electronic	+5 VDC SID 1 and SID 2	0.5 A	-

Table 16: Used Fuses

DISPLAY AND KEYBOARD

The 450-GC Touch Screen and Keyboard is an integrated user interface designed for maximum ease of learning and ease of use. This section describes the keyboard and display layout and functions.

on page 46



on page 54

on page 51

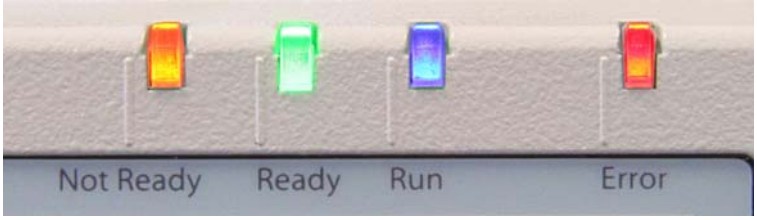
on page 48

on page 52

The 450-GC is carried out with a so-called touch screen. Touch simplifies the human / machine interface by enabling the operator reactions to be natural and intuitive. Varian touch solution also helps reduce the complexity of operations by eliminating the often cumbersome traditional operator interfaces. They simplify the task and reduce training time for operators while adding more capabilities to the machine itself.
Touching the display (with Stylus-Pen or Finger) will simplified the operation of the 450-GC.

STATUS INDICATION LED'S

The GC status indication LED's are situated in the upper right corner of the Keyboard/Display. The LED's will make it possible to monitor the GC status from some distance.



Overview Analytical Phases

Analytical Phase	*Status on Main Screen*	*LED Not Ready*	*LED Ready*	*LED Run*
Creation	Creating	ON	OFF	OFF
Initialization	Initialization	ON	OFF	OFF
Equilibration	Not Ready	ON	OFF	OFF
Stabilization	Stabilizing	BLINKING	OFF	OFF
Monitor	Monitor	BLINKING	OFF	OFF
Pre-Run	PreRun	BLINKING	OFF	OFF
Sample Preparation	Sample Prep	OFF	BLINKING	OFF
Ready	Ready	OFF	ON	OFF
Run - Hardware ready	Run	OFF	OFF	BLINKING
Run - Hardware Not Ready	Run / Not Ready	ON	OFF	BLINKING
Standby	Standby	ON	OFF	OFF
Fault	Error	ON	OFF	OFF

Table 17: Status Indication LED's

Error LED

For detailed error description on page 84

Error State	*Led Error*
Event	OFF
Alert	BLINKING
Error	ON

Table 18: Error LED

MOST USED FUNCTIONS

Throughout the push-buttons direct access is possible to the most used GC functions.

Main operation page, on page 54.

Status Instrument Page, on page 49.

Version information, on page 50 will show (detailed) firmware version information

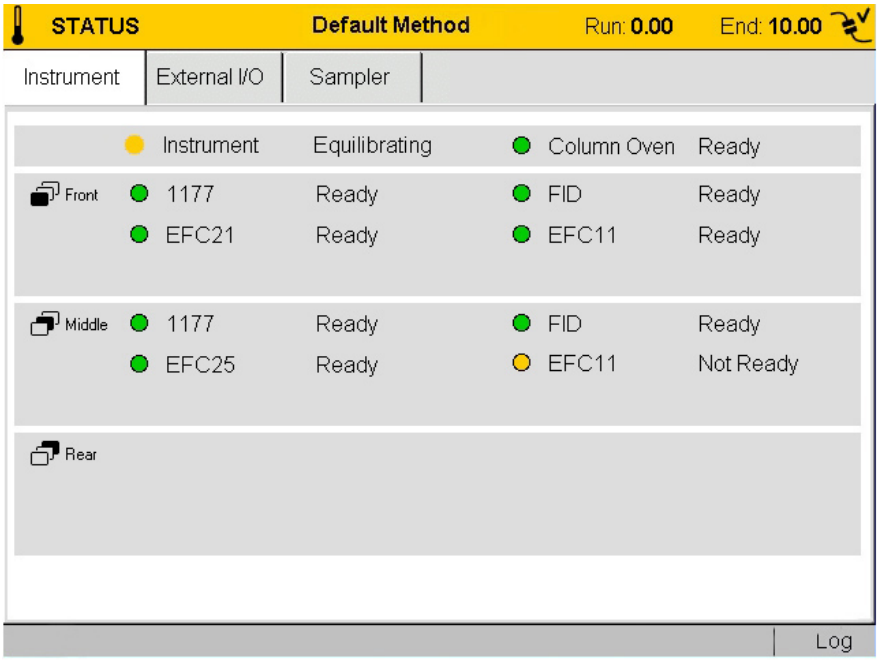
Start/Stop GC Run

Pause (Pause automation GC is running in automation mode, else ignored)

Status page

Allows the user to view the current status of the various components of the 450-GC.
It gives an overview about the status of the GC components, split up in the positions front, middle and rear.

All components visible are hyperlinked. Touching the display (with Stylus-Pen or Finger) in the area where more information/control is needed, the desired page will be shown.

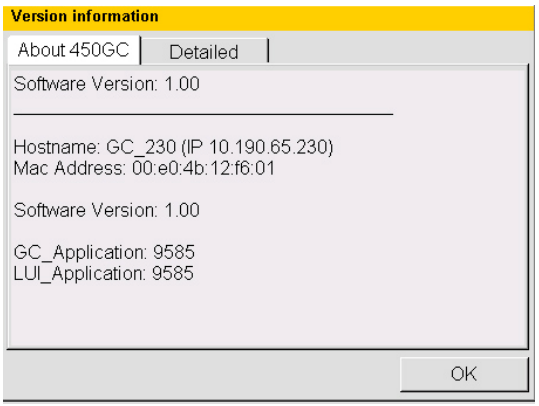


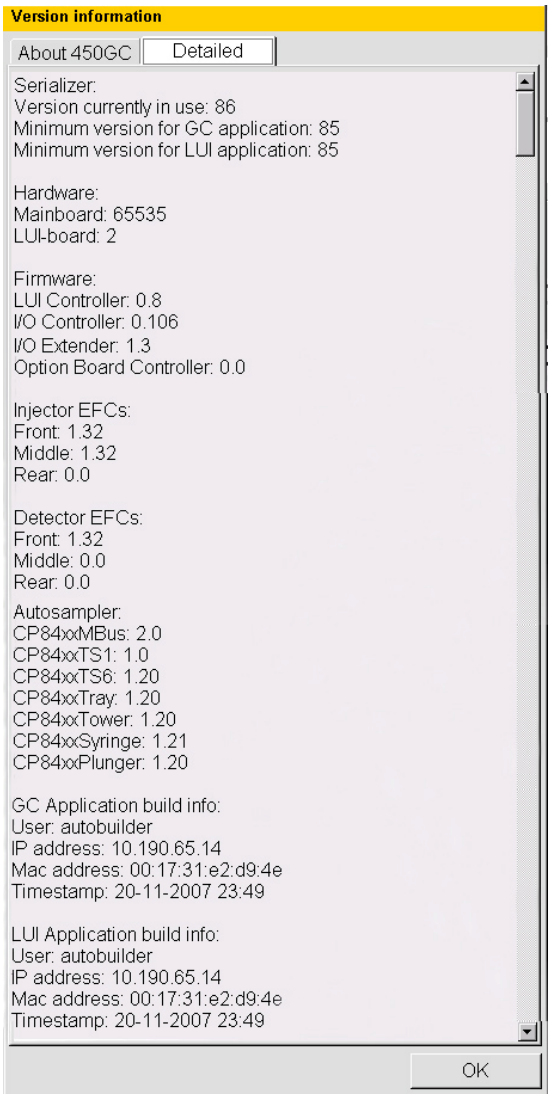
The colored dots indicate the status of the component:

- The green dot indicates the component is Ready.
- The yellow dot indicates the component is Not Ready.
- The empty dot indicates the component is switched off.
- The red dot indicates the component is in ERROR.

Version Information

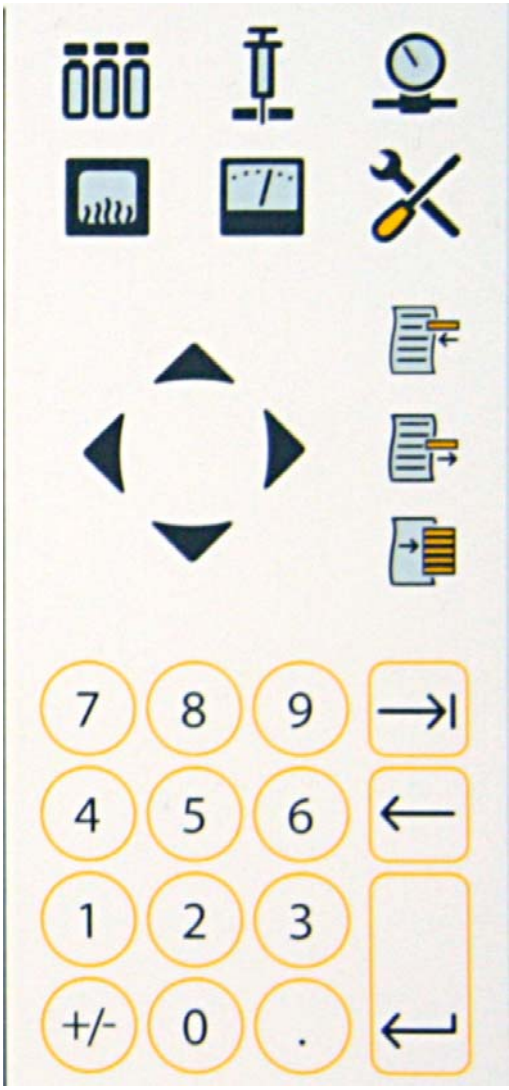
This screen will give information about the Software Versions used in the 450-GC. The “About 450GC” tab shows the Hostname and IP- and MAC address.
The “Detailed” tab give detailed firmware information about Hardware (main- and displayboard) Injector and detector EFCs, AutoSampler (if present) firmware.





ADVANCED FUNCTIONS

Throughout the keyboard direct access is possible to advanced GC functions.



Sample delivery section of the Sampler (and VLV-table), on page 55.

Injector screen, on page 57.

Pressure and Flow screen, on page 58.

Oven Screen, on page 60.

Detector screen, on page 62.

Setup/Calibration screen, on page 64.

Add line, adds a new line at the end of the table.
Insert line, adds a new line before the selected line.

Delete line, deletes the selected line in the table. When a line is deleted, then the table reformats to move the succeeding rows up.

Delete Table, clear all entries in the table except for the initial line.

Tab

Back space

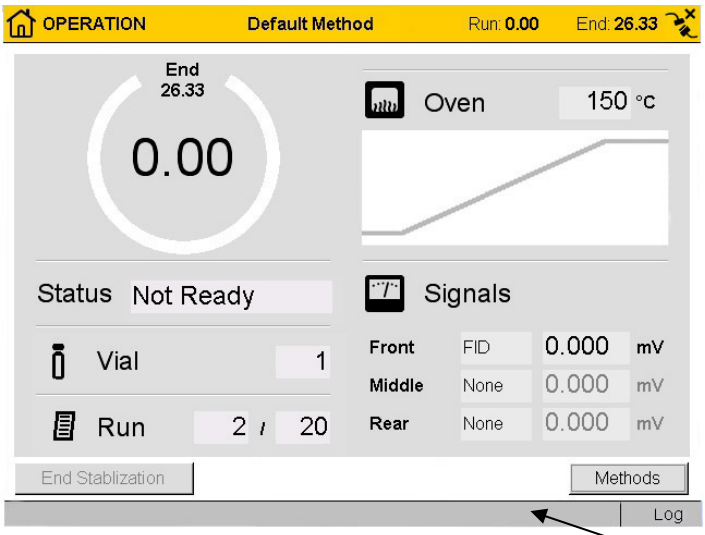
Enter

DISPLAY

The display is a LCD screen in combination with a touch-screen.
By touching the display (Stylus-Pen or finger) it is possible to control the GC.

The use of the Stylus-Pen is advised, it is more accurate and will keep the display free of fingerprints. The Stylus-Pen is located at the bottom right side of the Display/Keyboard, on page 45.

The display is split up in 4 sections:



« Title Bar, on page 53.

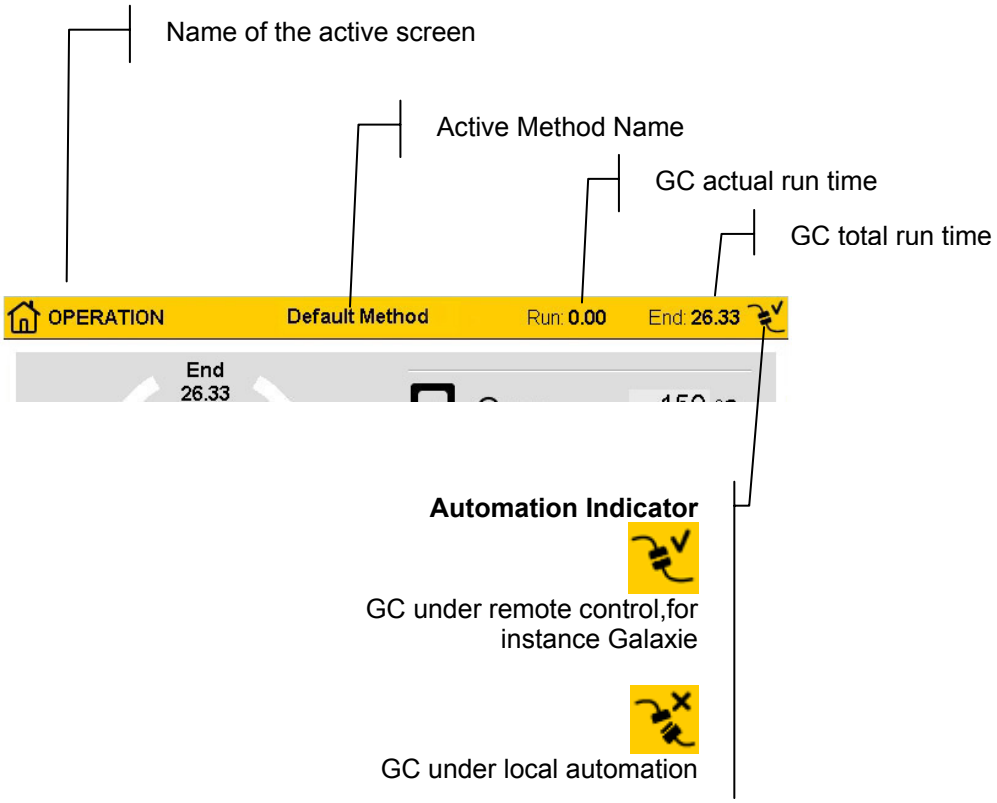
« Main Operation screen, on page 54

« Button Bar, in principle all buttons related to the active page will be visible in this bar.

« Message Bar, messages related to the active page or component will be displayed.

Title Bar

This bar is always visible with identical information, only the page name will change.

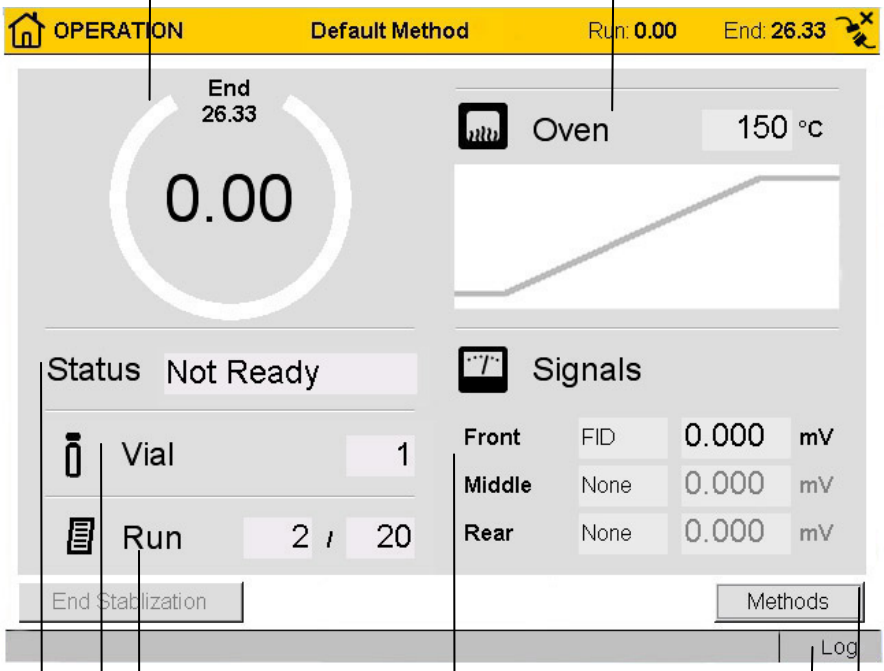


Main Operation screen

The main operation screen show the operation status, in which the most important parameters are visible.

Run indicator, fill up with blue when runtime is increasing. Touching the indicator will reverse the indicator. During stabilization the indicator will be yellow and an hour-glass will be visible.

Oven information, actual oven temperature and Oven ramping graphic is visible



Local Automation, run 2 of 20 is busy

Detector information, detector type and actual detector signal.

Sampler information, indicating run sample

Goto GC Log
Log
Needs customer attention on page 84

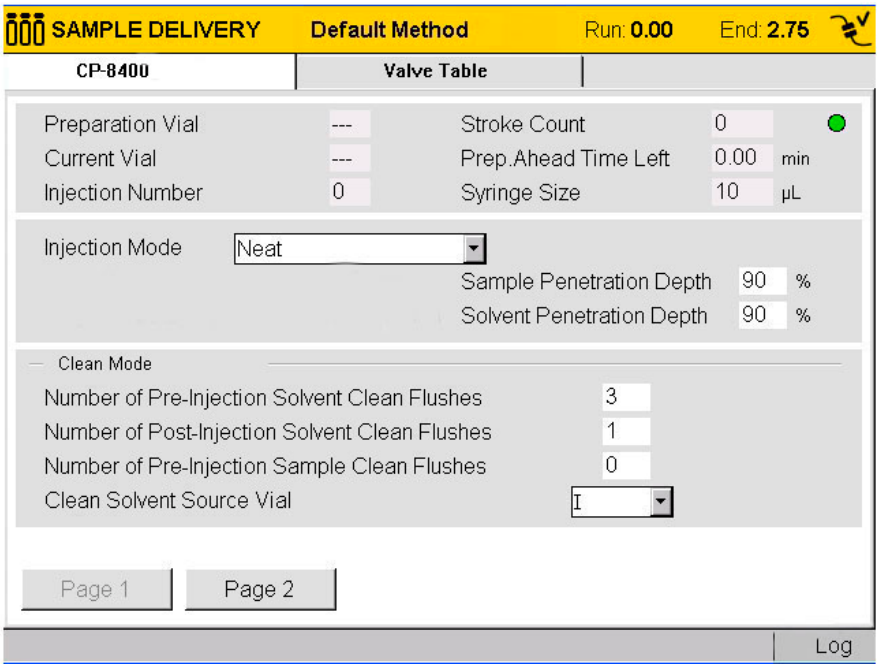
Status information, GC status identical as status indication LED's

Goto Methods page, method open, save page, on page 61

Sample Delivery Screen

Sample Delivery method parameters are accessible via the SampleDelivery key on the 450-GC keyboard. The Sample Delivery section can consists of multiple tabs, CP-8400/8410 AutoSampler, if present and Valve Table.

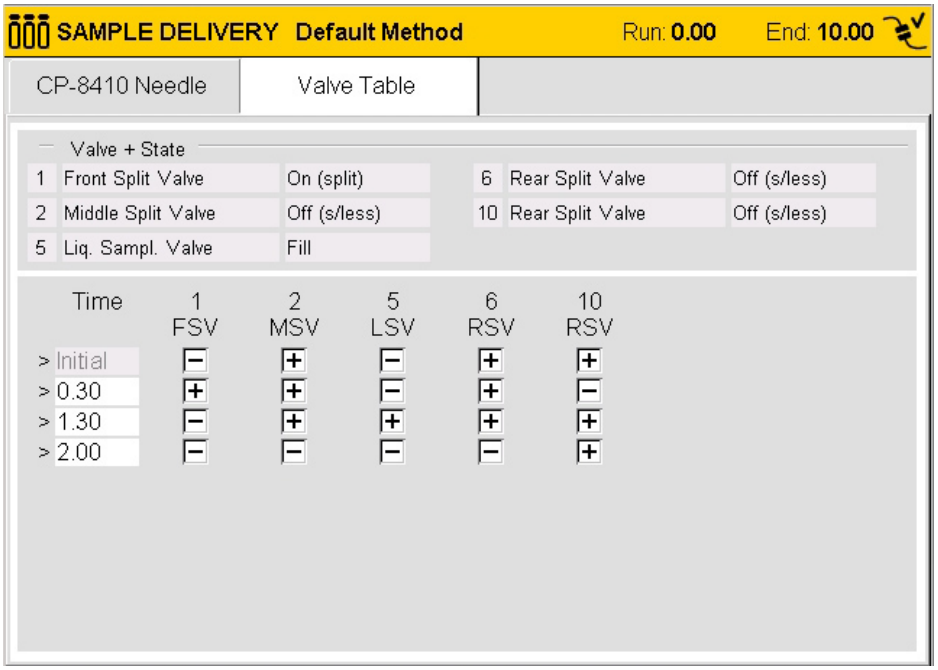
The CP-8400/8410 section have multiple pages represent the most frequently used parameters, however, predefined modes of operation limit the need to access all parameters. See Sample Delivery Parameters on page 123 for more information.



The Valve Table screen gives timed control over the valves (defined in the Setup/Sample Delivery).

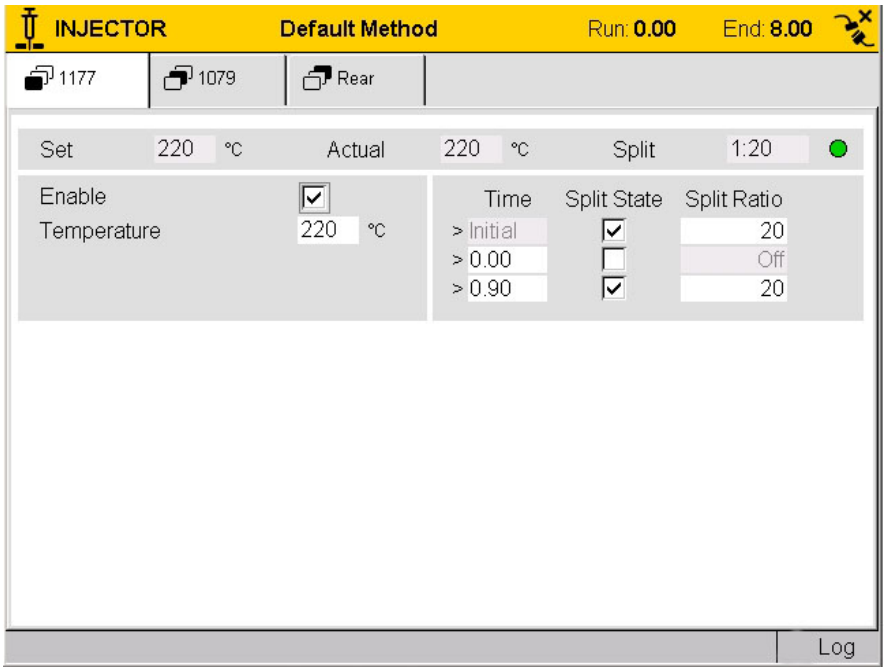
All valves are connected (hardware) to connector J42/J43 External events, on page 40 or to the option board connector J302/J30,on page 43.

The position of the valve is indicated by its energized (+) or de-energized (-) state.
Only the "enabled" (on page 71) valves are visible.



Injector Screen

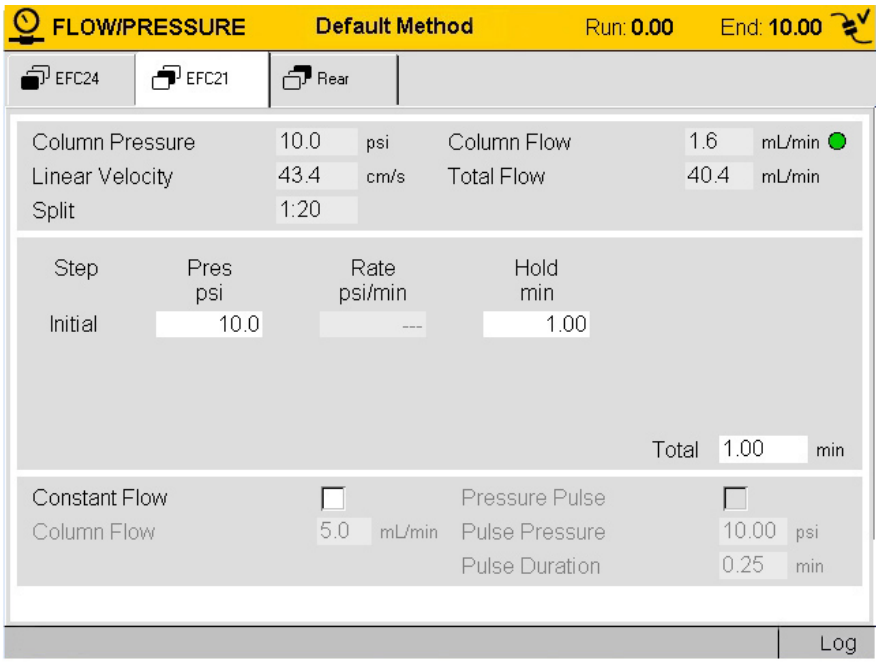
The INJECTOR screen allows you to modify the Injector parameters and view the status of individual Injectors. If more than one Injector is installed, pressing the corresponding tab will guide you to other Injector page.



The above example is for an 1177 Injector. Further details for programming Injectors can be found in the Injector screen on page 97 of this manual.

Pressure and Flow screen

EFC (Electronic Flow Control) method parameters and status are accessible via the FLOW/PRESSURE key on the 450-GC keyboard. If more than one EFC module is installed, pressing the corresponding tab will guide you to specific EFC page.



EFC modules are identified by type (EFC21 in this example) and location (front, middle, rear) to coincide with the sample delivery or injection device with which they are associated. Type EFC21 is used with the 1177 or 1079 PTV. The above screen is a typical EFC program for type EFC21. In this case, a pressure program is applied to keep the column flow rate constant while the column temperature is ramped.

Four types of Injector EFC modules exist and each type is associated with a specific GC Injector system.
The Setup menu identifies each EFC type and its associated Injector system by location In Table 19, on page 59 is a description of each EFC type with used Injector.

EFC21 setup calibration (1177 and 1079 PTV Injectors) also contains a Septum Purge Calibration button and Splitless Vent Flow.

Septum Purge Calibration involves measuring a flow rate at a set pressure and entering those values into the system to correctly calculate the settings for split and column flow. The Splitless Vent Flow provides a vent purge for runs in which the split valve remains closed for the duration of the run. It should be set to at least 20 mL/min.

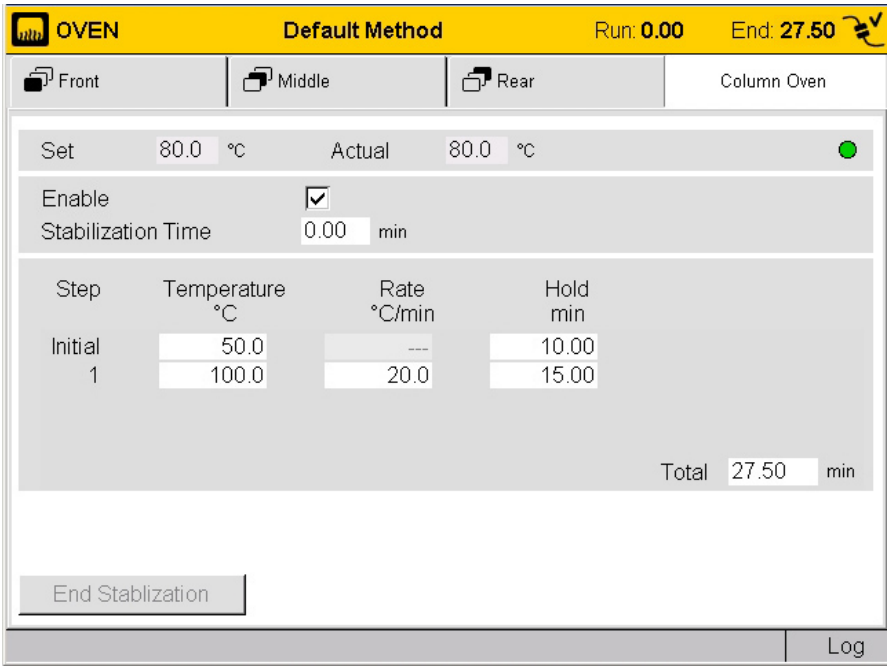
Sample Introduction System	***EFC Module***	***Optional Pneumatics System***
1177	EFC21	Manual pneumatics - inlet flow controller/back pressure regulator with pressure gauge.
1079 PTV	EFC21	Manual pneumatics - inlet flow controller/back pressure regulator with pressure gauge.
1041 Injector	EFC23	Manual digital flow controller with pressure gauge
1041 Injector	EFC24	Manual pressure regulator with gauge.
1061 Injector	EFC23	Manual digital flow controller with pressure gauge
1061 Injector	EFC24	Manual pressure regulator with gauge.
Gas or Liquid Sampling Valves	EFC24	Manual pressure regulator with gauge.
Purge and Trap with either 1177 or 1079 PTV	EFC25	Manual pneumatics - inlet flow controller/backpressure regulator with pressure gauge.
Headspace with either 1177 or 1079 PTV	EFC25	Manual pneumatics - inlet flow controller/back pressure regulator with pressure gauge.
1093 Injector (SPI)	EFC23	Manual digital flow controller with pressure gauge

Table 19: Injector EFC Modules

Oven Screen

The OVEN screen allows you to establish the column oven temperature, build a temperature program, or view the column oven status.
The status field for the OVEN indicates the programmed set temperature, the actual measured temperature and whether the column oven is ready, not ready or in error (colored dot).

Similar to other programming tables, the initial or isothermal line will always appear on the first line of the table. You can then build a temperature program by adding new lines. Below is a column oven program with an initial temperature, initial hold time, programmed ramp and final hold. The ranges of the Column Oven temperature are dynamically depending on injectors, detectors, columns and cryo options.
Stabilization time is the period the column oven will equilibrate for before allowing the column oven to go READY. It will count down (visible in the Main Operation screen) from the set value to 0.00 min when the stabilization period is over. The Oven Power checkbox is used to turn the column oven heater and fan ON or OFF.
The *End Stabilization button* is used to bypass the remainder of the stabilization time.



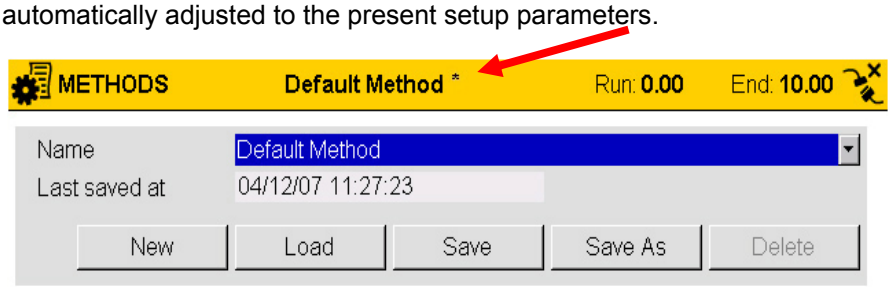
Methods Screen

By the method screen it is possible to load, save, save as, create (new) and delete methods.
During first (after factory) startup a Default Method will be loaded, all heated zones will be set at 50°C.

The top line of the screen identifies the method that is currently active, the current run time and the end time of the active method and whether the GC is under local or remote control.

The "*" behind the method name is an indication that the present method parameters are different then one saved (example: initial oven temperature has been changed manually).
Saving the method under the same or other name will let the "*" disappear.

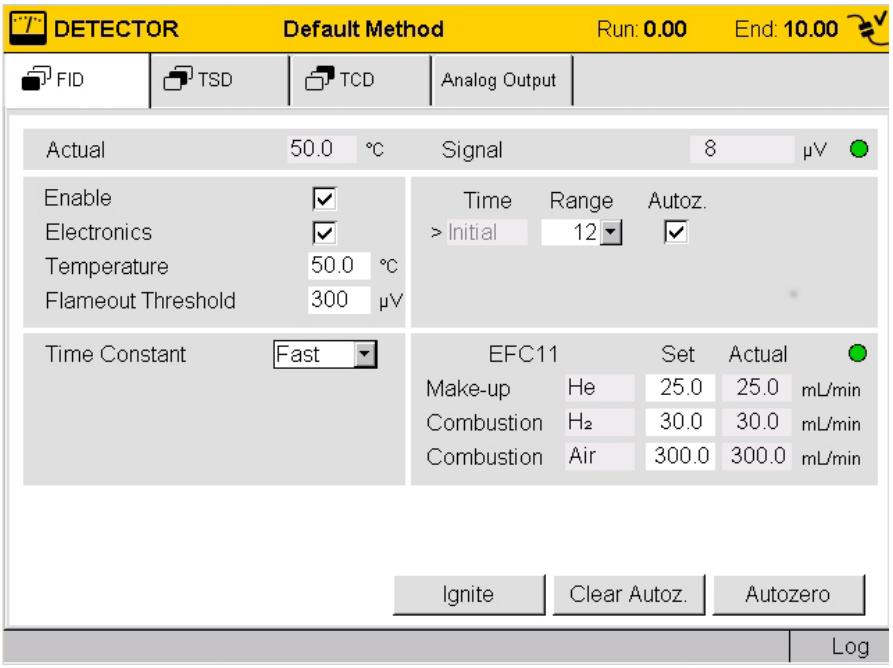
The "*" will also disappear when the loaded method parameters are in conflict with the setup parameters. Example: the maximum column temperature in the setup screen is changed (other column used), if a (older) method is loaded the system will detect the difference in maximum column temperature and let the "*" appear. Saving the method again will let the "*" disappear. The method will be automatically adjusted to the present setup parameters.



Touching the Method name in the Title Bar will switch to the Method screen.

Detector Screen

The DETECTOR screen allows you to modify the detector method parameters and view the status of individual detectors. If more than one detector is installed, pressing the corresponding tab will guide you to other detector screens. If the option Analog Output is installed a tab Analog Output on page 63 will be visible.



The above example is for an FID detector. Further details for programming detectors can be found in the detector screen on page 279 of this manual.

Six types of detector EFC modules exist and each type is associated with a specific GC detector system.

In Table 20, on page 63 is a description of each EFC type with the used detector.

Detector Type	*EFC Module*	*Optional Pneumatics System*
FID (Flame Ionization Detector)	Type EFC11	**Hydrogen:** Valve 0-50 mL/min **Make-up:** Valve 0-55 mL/min **Air:** Valve 0-550 mL/min
TSD (Thermionic Specific Detector)	Type EFC12	**Hydrogen:** Flow controller 0-6 mL/min **Make-up:** Valve 0-55 mL/min **Air:** Valve 0-200 mL/min
ECD (Electron Capture Detector)	Type EFC13	**Make-up:** Valve 0-60 mL/min
TCD (Thermal Conductivity Detector)	Type EFC14/16	**Make-up:** TCD Make-up EFC14: 0 – 60 mL/min (2x) **Reference:** TCD Reference EFC16: makeup: 0 – 60 mL/min Ref: 0 – 55 mL/min
PFPD (Pulsed Flame Photometric Detector)	Type EFC15	**Hydrogen:** Flow controller 0-33 mL/min **Air 1**: Flow controller 0-33 mL/min **Air 2**: Flow controller 0-55 mL/min

Table 20: Detector EFC Modules

Analog Output

For detailed description of the analog outputs click here, on page 280.

Setup/Calibration

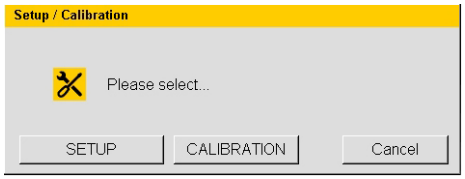
The Instrument SETUP key allows access to the instrument configuration screens for the purpose of viewing or modifying the 450-GC configuration, it also allows calibrating parts of the GC hardware.

The 450-GC has already been configured at the factory and requires only minor input from the user.

Access to SETUP screen is only possible if:

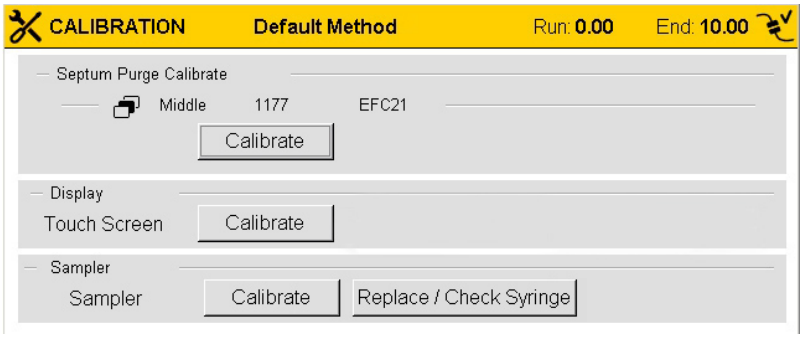
- ***GC in not in run state***
- ***No error(s) are present (in case of an error, goto error log on page 84 and "reset error" before continue.***

Press the SETUP key and select: Setup or Calibration



Calibration

The Calibration page will appear:

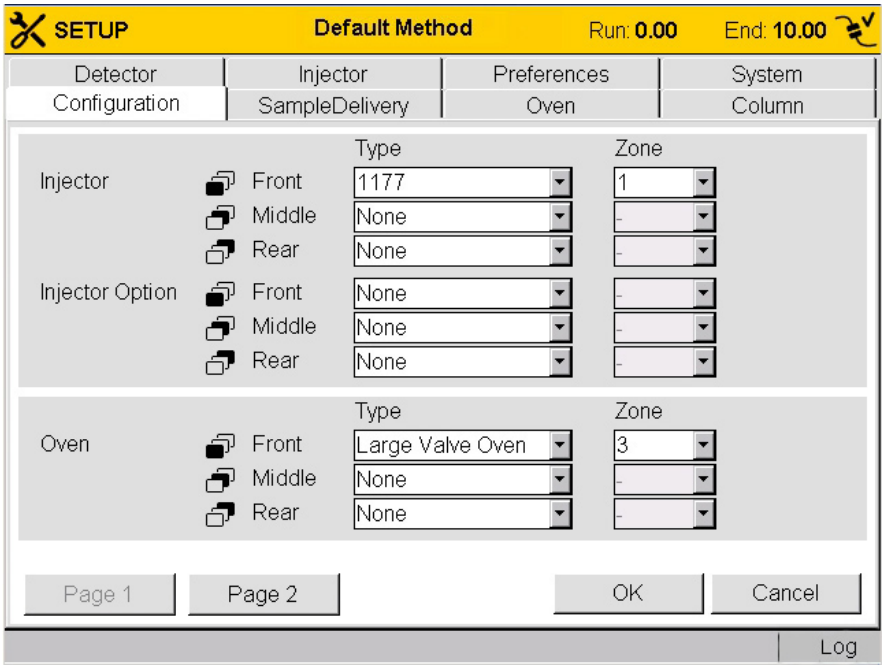


The next calibration possibilities can appear:

- Septum Purge Calibration of the Injectors: 1177 on page 165 and 1079 on page 188.
- Display touch screen calibration on page 81.
- Sampler Calibrate on page 112 and Change Syringe on page 119.

Setup

At the time of installation, the SETUP screen is normally accessed by the user to check/update the local time and date information, update column parameters, or verify the instrument configuration. If the instrument configuration is changed, such as when adding a field upgrade kit, the involved relevant instrument SETUP screen **must** be updated.



More details concerning the Setup Tabs just click on the links below:

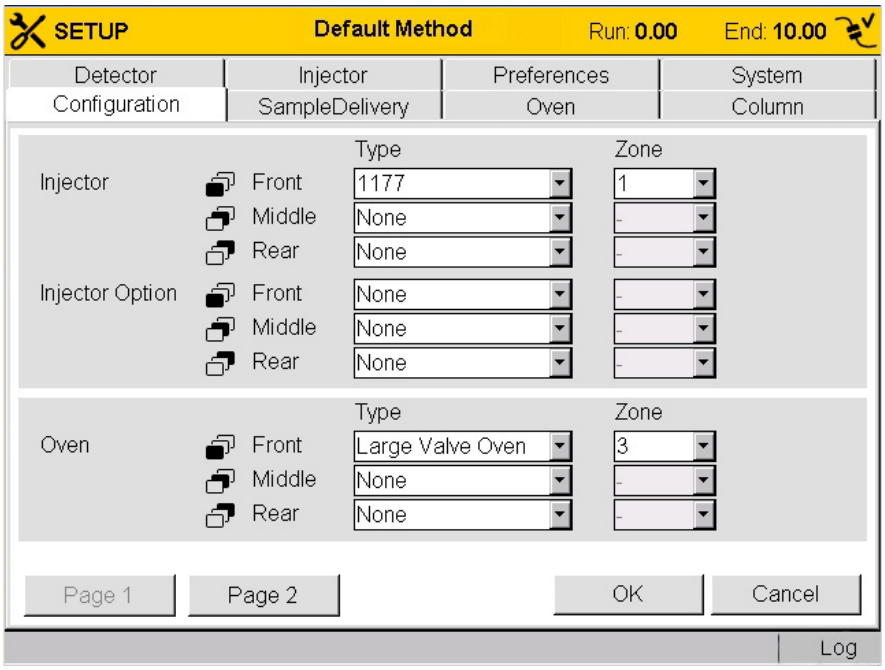
Configuration Tab on page 66, Sample Delivery Tab on page 68,
Oven Tab on page 73, Column Tab on page 74,
Detector Tab on page 75, Injector Tab on page 77,
Preferences Tab on page 78, System Tab on page 79

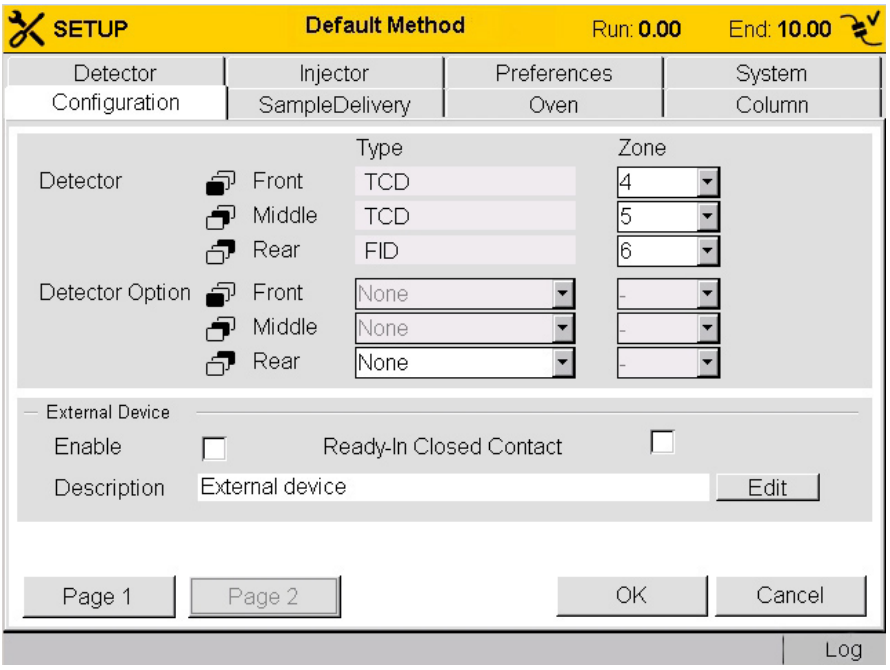
Configuration Tab

In the Configuration Tab it is possible to set the type of injector, Injector options and Oven in relation with each of the heated zones (their physical location) of the 450-GC.

The Column oven always occupies 1 heated zone (zone 7). The other available heated zones are numbered 1 through 6 and can be occupied by 1 of the following devices: 1177 Injector, 1079 PTV Injector, 1041 Injector, 1061 Injector, 1093 Injector, SPT, FID, TCD, ECD, TSD, PFPD, large valve oven, small valve oven, or methanizer. SPT is allowed only in zone 3. Dual TCD is using 1 heated zone for 2 detectors.

The location typically refers to whether the device is installed in the front, middle, or rear position on the top of the 450-GC. In the case of detectors the location also refers to the relative positions of the detector electrometers installed on the main electronics board. By default, zones 1, 2 and 3 are identified as front, middle and rear and zones 4, 5 and 6 are identified as front, middle and rear.
Zones 1, 2 and 3 are normally used for Injectors and zones 4, 5 and 6 are used for detectors.





External Device

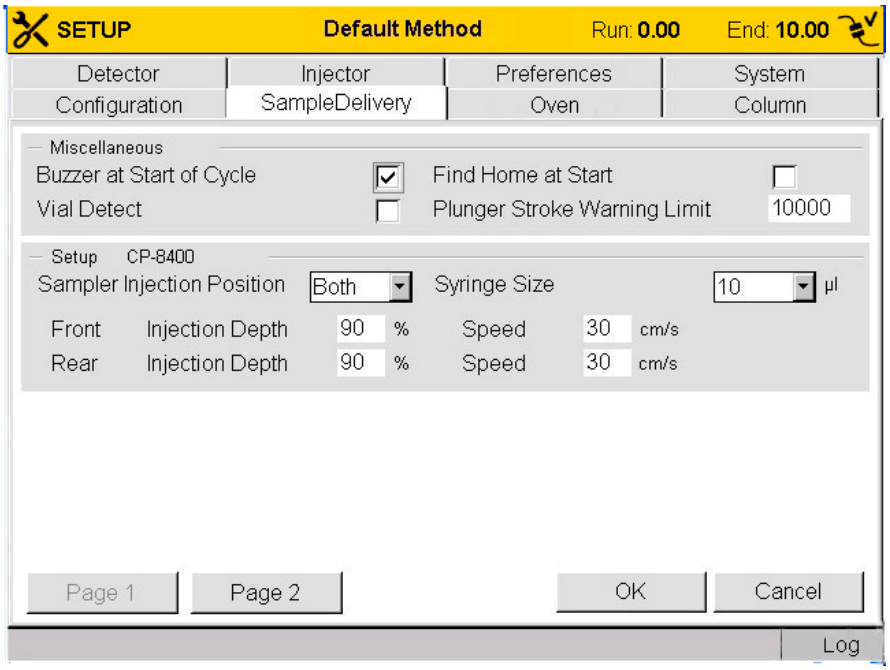
If an external device is connected that needs to be ready prior GC start, the GC state (ready, not ready) can include the state of the external device.

When the external device state is included a selection must be made if the ready-in (pin-1 on connector J401 Analog Out on page 36) of the GC is normally closed or normally open.

External device contact	*Enable check box*	*Ready-In check box*
Normal Closed contact (N.C.)	√	√
Normal Open contact (N.O.)	√	-
Exclude ext. device	-	Grayed out

Sample Delivery Tab

This tab has Setup parameters for an AutoSampler, if installed (Page 1) and Valving (Page 2).
Before calibration/operation of the sampler it is advised to check the parameters on this screen.



The following AutoSampler parameters are visible:

Miscellaneous screen

Buzzer at Start of Cycle – A sound will be generated to warn of impending tower movements when this parameter is checked. The default value is checked.

Vial Detect – Checked will cause the CP-8400/8410 to halt and generate an error if a missing vial is encountered. Unchecked will cause the AutoSampler / AutoInjector to ignore a missing vial and continue until it finds a vial within the specified range. The default value is unchecked.

Find Home at Start – Checked will cause the CP-8400/8410 to find its home position before starting automation or before an autoinject action. If several people are using the 450-GC on which the AutoSampler / AutoInjector is mounted, then it is advisable to leave this setting checked. Use the unchecked selection for higher throughput.

Plunger Stroke Warning Limit (1-100,000) – This parameter defines the expected number of strokes the plunger is expected to survive without significant degradation. Once the entered value is exceeded, a message will be displayed in the error log screen indicating the plunger counter has exceeded the set value.

The default number of strokes is 10,000.

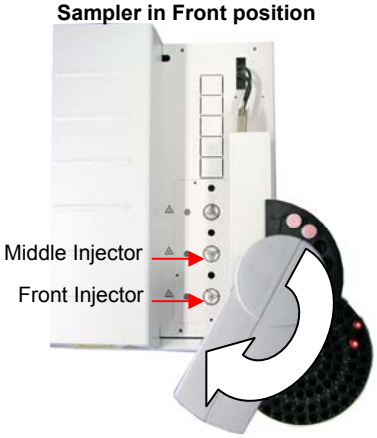
Setup (CP-8400)

Sampler Injection Position (Both/Front/Rear)
The CP-8400/8410 is mounted in either the front or rear position. The sampler position determines which of the 2 Injectors are reachable by the sampler.

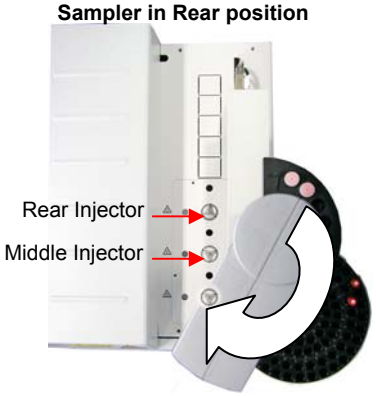
Sampler mounting	*Injector position*
Front	Front
	Middle
Rear	Middle
	Rear

Table 21: Sampler/Injector position

Sampler in Front position



Sampler in Rear position



In case the sampler is mounted in the front position it can inject in the front and middle Injector. In case the sampler is mounted in the rear position it can inject in the middle and rear Injector. In the sample delivery page of Setup, the front sampler Injection position is the first Injector (clock wise rotation of the sampler) the sampler is able to reach. The rear sampler Injection position is the second Injector the sampler is able to reach.

If 1 Injector is installed, make certain to specify the correct Injector. An incorrect setting may cause the CP-8400/8410 to inject into the top cover resulting in

damage to the syringe and the top cover. Select both setting if you have 2 Injectors that you wish to use with the sampler. Note that selecting both allows the sampler to use 2 Injector volumes when dual/duplicate mode is used. If you configure a single Injector, you will see a single injection volume in the automation tables.

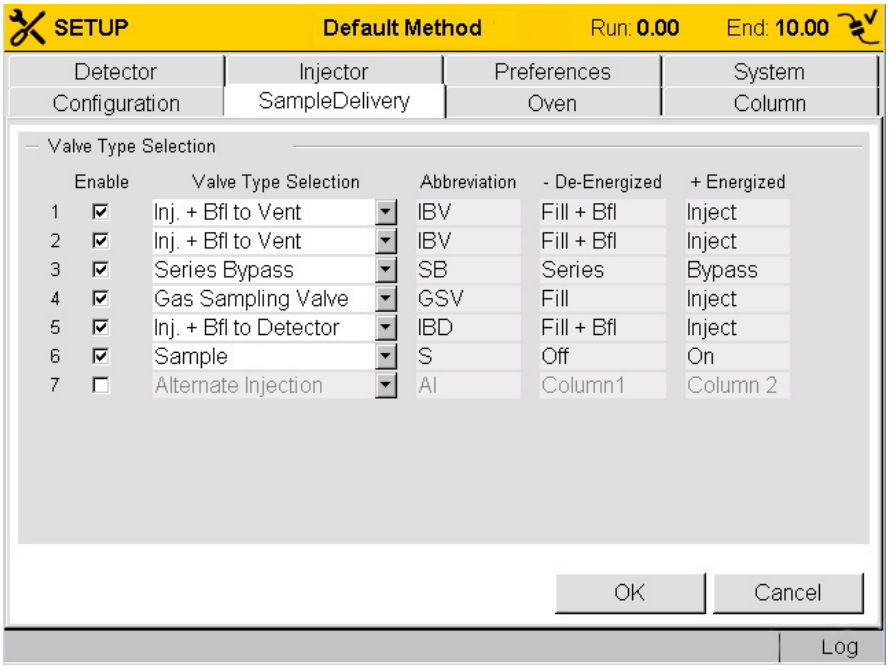
Syringe Size (1 μL, 2μL, 5 μL, 10 μL, 50 μL, 100 μL and 250 μL). (Note that this field is not visible when the AutoSampler is in the SPME mode). Enter the syringe volume you will install and use with your CP-8400/8410. The 450-GC will use this value to set the correct entry selections and any needed internal settings to correctly use the indicated syringe size.
If you are simply changing a syringe with a different volume syringe you can press “OK” and Exit at this point to accept the new syringe size.

Front/Rear Injection Depth (0-100 %). The Injector Needle Depth parameters define how far into the designated Injector the needle will penetrate. 100% causes the needle to be completely inserted to its 2-inch length. The valid range is settable in 1% steps. The default is 90%.

Front/Rear Speed (0-100 cm/s) The Injector Injection Speed parameters define the rate at which the needle will penetrate the Injector septum and descend to the set Injector Needle Depth. The valid range is settable in 1 steps. The default is 30 cm/s.

Valving

This screen configures the 7 standard external events available on the 450-GC that are typically used to control the 1177 and 1079 split valve with manual pneumatics (non-EFC) or actuators for sampling or switching valves. Each valve is identified in SETUP by a name that indicates its application. When a name is chosen this also defines the default states (Abbreviation, - De-Energized and + Energized) of the valve.
The Option board has 8 optional external events available and will be numbered from 8 till 15.
Only valves that are enabled are shown in the method pages.



In Table 22 the complete set of valve names and associated states that are available for programming.

Valve Type	*Abbreviation*	*De-Energized State (-)*	*Energized State (+)*
Front Split Valve	FSV	ON (split)	OFF (s/less)
Middle Split Valve	MSV	ON (split)	OFF (s/less)
Rear Split Valve	RSV	ON (split)	OFF (s/less)
Gas Sampling Valve	GSV	Fill	Inject
Liquid Sampling Valve	LSV	Fill	Inject
Sample	S	OFF	ON
Internal Standard	IS	OFF	ON
Surrogate Standard	SS	OFF	ON
Series Bypass	SB	Series	Bypass
Backflush to Detector	BD	Forward	Backflush
Backflush to Vent	BV	Forward	Backflush
Column Selection	CS	Column 1	Column 2
Injection + Backflush to Detector	IBD	Fill + Backflush	Inject
Injection + Backflush to Vent	IBV	Fill + Backflush	Inject
Alternate Injection	AI	Column 1	Column 2
Simultaneous Injection	SI	Fill	Inject
Methanizer Bypass	MB	Series	Bypass
Sample Preconcentration Trap	SPT	SPT Desorb	SPT Trap
External Event A	EA	OFF	ON
External Event B	EB	OFF	ON
External Event C	EC	OFF	ON
External Event D	ED	OFF	ON
External Event E	EE	OFF	ON
External Event F	EF	OFF	ON
External Event G	EG	OFF	ON
External Event H	EH	OFF	ON
External Event I	EI	OFF	ON
External Event J	EJ	OFF	ON
Power Fail Option Solenoid	PFOS	OFF	ON
Quick Switch Valve	QSV	OFF	ON

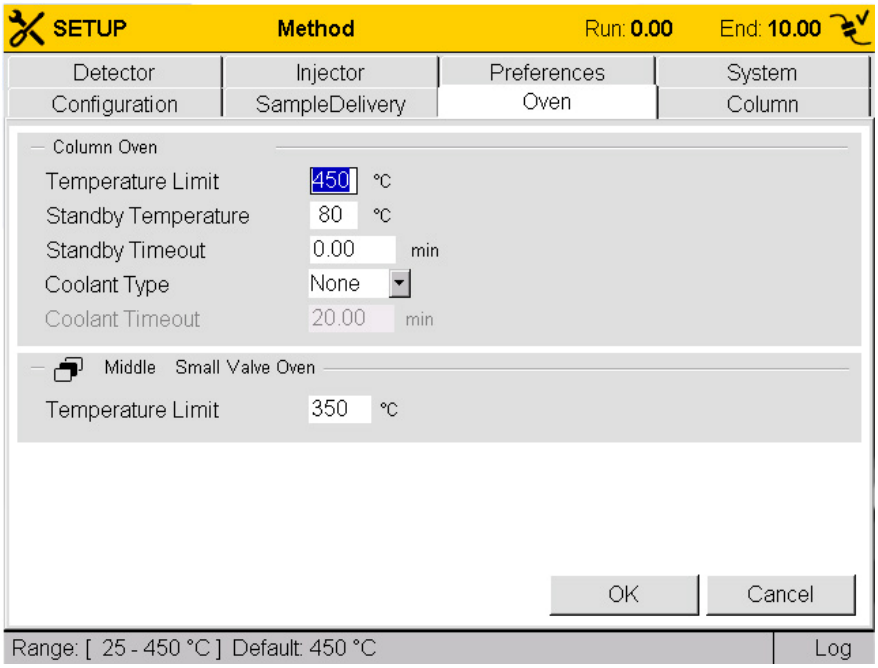
Table 22: Valve Type Names

Oven Tab

The default temperature limit of the column oven is set at 450°C (If the column limit is set lower then 450 °C the oven limit is set accordantly, the column with the lowest column temperature limit is used as maximum temperature limit), but can be set to a lower value.
If the 450-GC is idle for more then the Standby Timeout time, the column oven temperature is set to the Standby Temperature and 450-GC will go into Standby mode, see operation screen. This standby temperature will be invoked after the timer counts to zero, provided another run is not started before the timer has timed out.
The default Standby Temperature is 60°C.
The default Standby Timeout time is 120 minutes.
A value of 0 minutes in Standby Timeout will disable the Standby Temperature.
The Coolant checkbox turns on or off the cryogenic supply for the oven, if installed. Coolant type can be LN_2 or LCO_2..
Coolant time-out refers to the period of time for which the coolant will remain on after it is turned on. For example, if the set temperature has not been reached or the run has not started within the specified time, the coolant supply will be turned off.

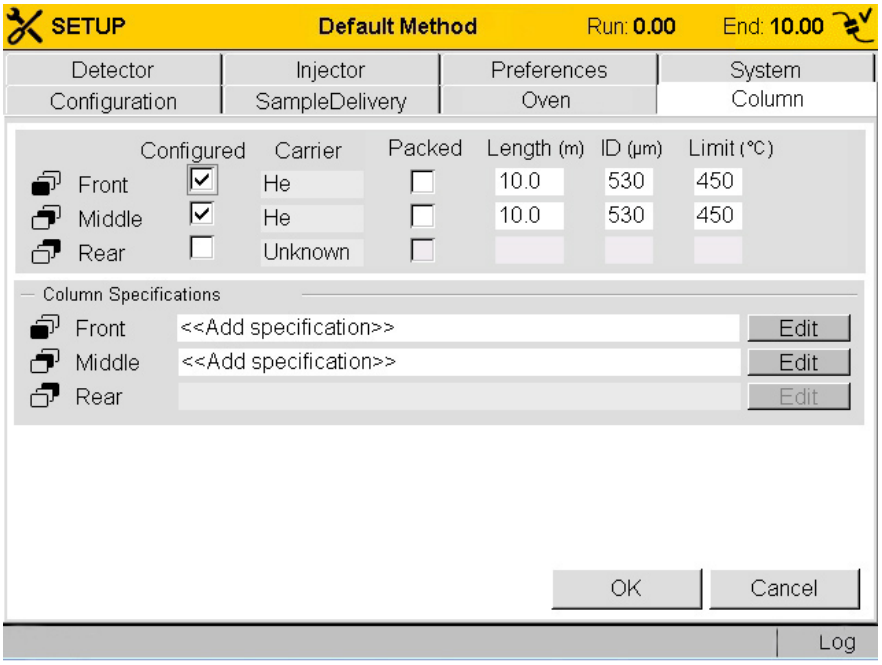
WARNING

Leave always the Injector(s) / Detector(s) heating ON (enable) and at a temperature of at least 100 °C when using cryogenic column oven cooling!



Column Tab

Column parameters such as packed column, column length, internal diameter and choice of carrier gas are used for the flow calculations of EFC.
The column limit temperature(s) have a direct relation with the maximum oven temperature. If set at 350 °C the oven temperature limit will be also 350 °C.
The column with the lowest column temperature limit is used as maximum temperature limit.
Carrier gas is configured by carrier gas selected for the corresponding Injector.
In case of a packed column the parameters column length and internal diameter are not used.



Detector Tab

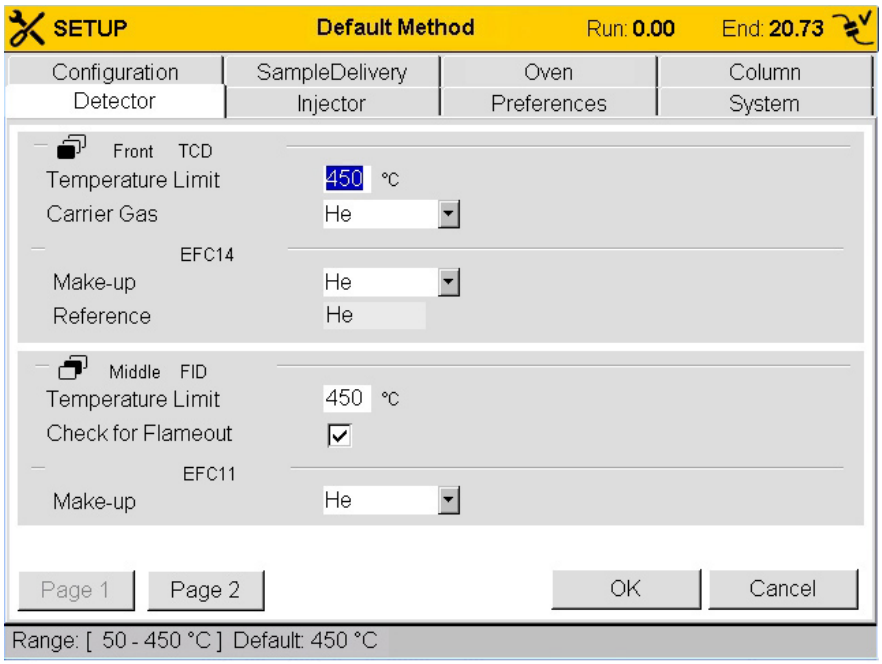
The detector tab identifies each Detector system and its associated EFC type detector system by location (front, middle, rear). In case more information is available this will be shown on next pages. The last page (in example Page 2) Analog output is shown.

In Table 20, on page 63 is a description of each EFC type with the related detector.

Detector EFC involves selecting the relevant carrier/make-up/reference gas type.

The default temperature limit of all detectors is set at 450°C, but can be set to a lower value.

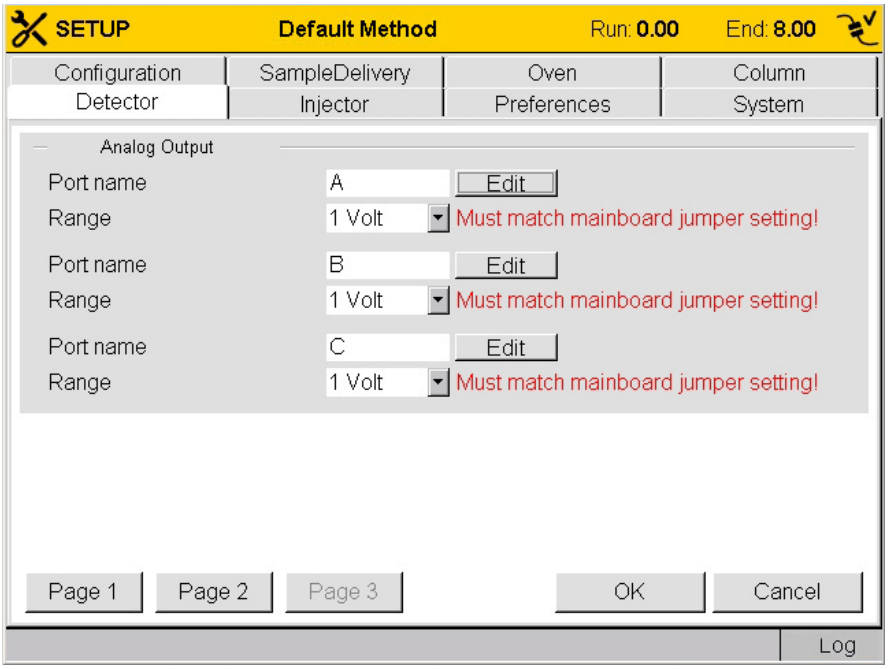
The 450-GC monitors the FID background current continuously and will attempt to light the flame (only if the checkbox “Check for Flameout” is checked) if the current drops below a specified threshold and the detector temperature is still above 100 °C (for all Ignite conditions click here on page 287. Up to three attempts to ignite the flame will be made before the 450-GC reports a flame-out fault.



On the second page (Page 2 or Page 3) the Detector Analog Output screen will be visible (only if the Analog option board is present).

The Port name can be changed by the button “Edit”.
The Port name will be used in the Detector screen, on page 280.

If the “Range” setting must be changed, make sure that the Range setting matches the mainboard jumper settings!



Injector Tab

The Setup menu identifies each Injector system and its associated EFC type detector system by location (front 🗗, middle 🗗, rear 🗗).

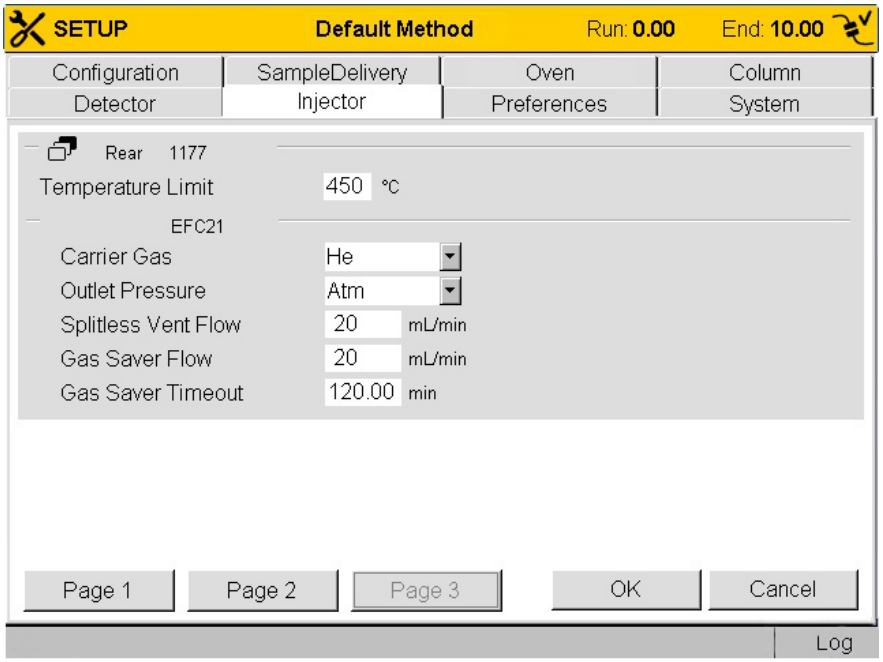
The default temperature limit of all Injectors is set at 450°C, but can be set to a lower value.

Injector EFC involves selecting the relevant carrier gas/outlet Pressure and more parameters.

In Table 19, on page 59 is a description of each EFC type with used Injector.

Outlet Pressure is used to distinguish between a column venting to atmosphere or a column venting to a vacuum source such as a mass spectrometer.
A specific Gas Saver Flow and a timeout period may be set. **A time entry of 0 turns the Gas Saver feature off**. If a run is not started or setup activated before the timer expires, the set Gas Saver flow rate will be started. A message will be logged in the instrument log.

The Splitless Vent Flow is the flow used for calculating the total flow when the method split program contains only splitless events.

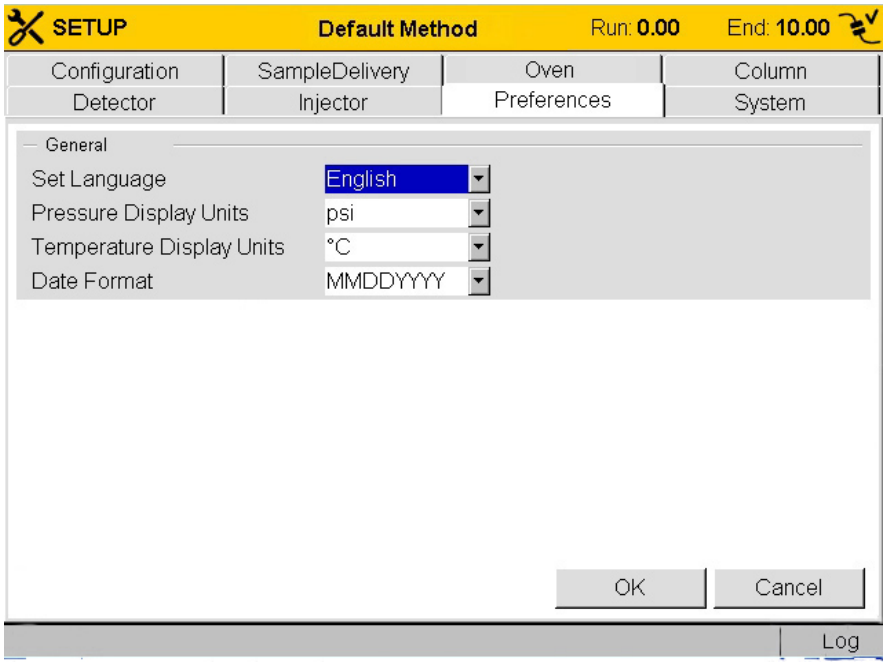


Preferences Tab

In this tab it is possible to set General parameters.

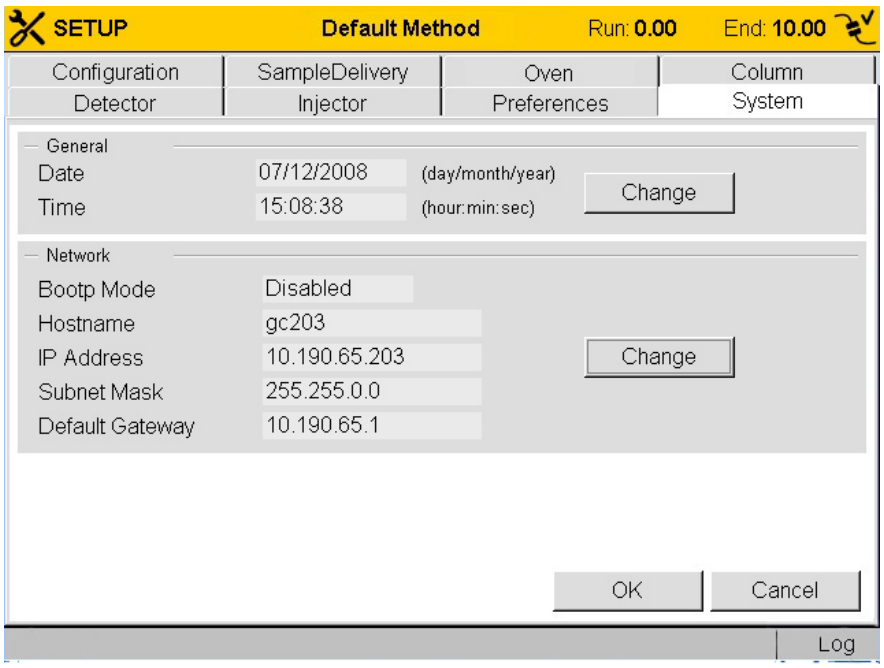
General:

- Set Language, default English.
- Pressure Display Units, default psi also kPa and bar possible.
- Temperature Display Units, default °C also °F possible.
- Date Format, default MMDDYYYY, also DDMMYYYY, DDMMYY and MMDDYY possible.



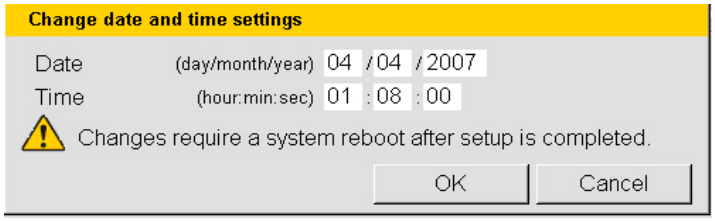
System Tab

In System tab it is possible to set General and Network parameters.
The Network Change button is un-usable when the network-switch at the Rear panel, on page 42 is set at DHCP[a] (BOOTP).



General:

Date and Time, pressing the change button, it is possible to change both values.



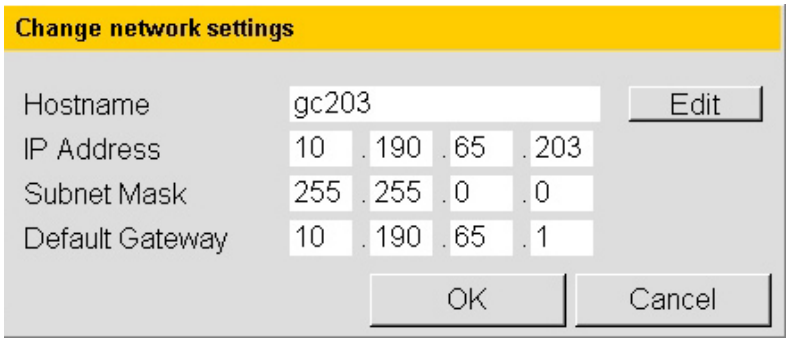
After changing the Date and Time a reboot is required. The OK button of the Setup screen will change to "OK reboot". Rebooting is performed automatically after pressing the "OK reboot" button.

[a] DHCP is used to obtain an IP address from Galaxie Workstation, on page 90 or a server.

Network:

Network, pressing the edit button it is possible to change network settings.
It's possible to set a fixed IP address in the 450-GC.
Check before changing the network settings if on the Rear panel, on page 42 the dip-switch is set to the "Fixed IP" position. If not set switch in "Fixed IP" position and restart the GC.

Fill in the IP address and Hostname, make sure the Subnet Mask and default Gateway are compatible with the PC settings.



Touch Screen Calibration

Description

Calibration is necessary because it is difficult to perfectly align a touch screen's coordinates to the LCD display behind it. If a button or other "live" feature on the display is to be properly activated, the coordinates of the area touched on the screen must be sufficiently close to the coordinates of the feature on the display. Otherwise, the software may not correctly act upon the soft button presses.

The Touch-screen is factory calibrated, however re-calibration of the touch-screen can be required (possible reasons: viewing angle, temperature or humidity changes or after vibration or shock).

Calibration

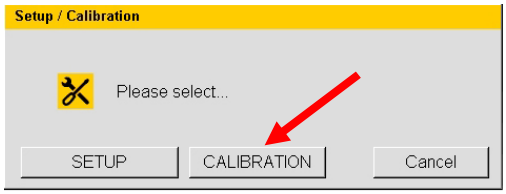
Carried out the touch-screen calibration only with the Stylus-Pen.

CAUTION

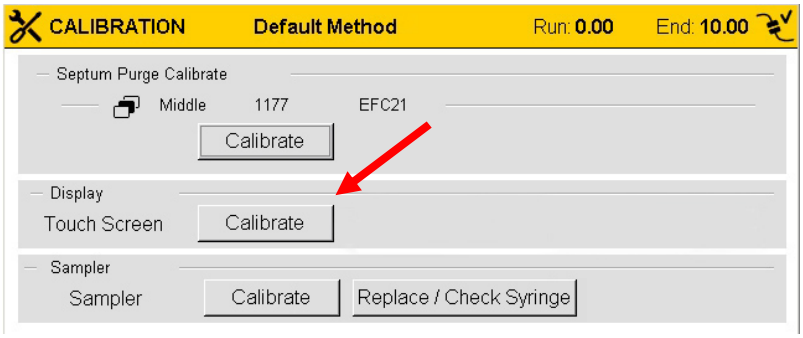
Do not touch the display during the calibration procedure!
This can result in an in-correct calibration.

It is possible to navigate to the touch-screen calibration procedure (on page 83) using only the hardware keys.

Press the SETUP key and select: Calibration



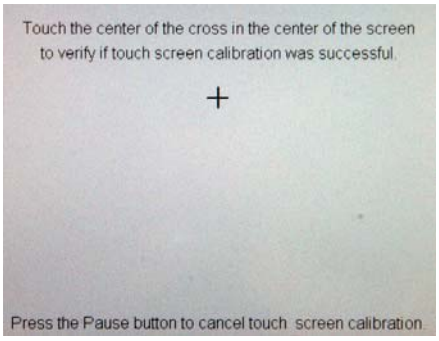
The Calibration page will appear:



Press in the screen Display, Touch Screen the button “Calibrate”.

The procedure will ask to touch (using the Stylus-Pen!) a number of calibration points (cross in Upper left and right, lower left and right corners) visible on the display.

Press at any time the Pause button to cancel the calibration.

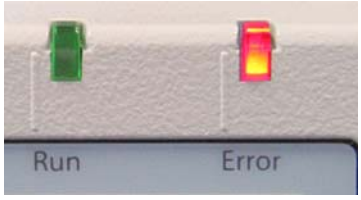


Final, as verification, touch the center of the cross in the center of the screen. If the calibration results are accepted by the software, press the “Start/stop” button to exit the calibration screen.

If the touch screen is not accessible for screen calibration, it is possible to access via the hard-keys.

1. Press the question mark hard-key, this will bring up the Version Information screen.
2. Press the following hard-keys in the correct order (not to fast!):
3. Use the correct sequence, please note that the (push/wait) timing is important.
4. Try several times if the calibration screen will not directly popup.
5. Follow the instruction on the calibration screen.

Error/Log Handling

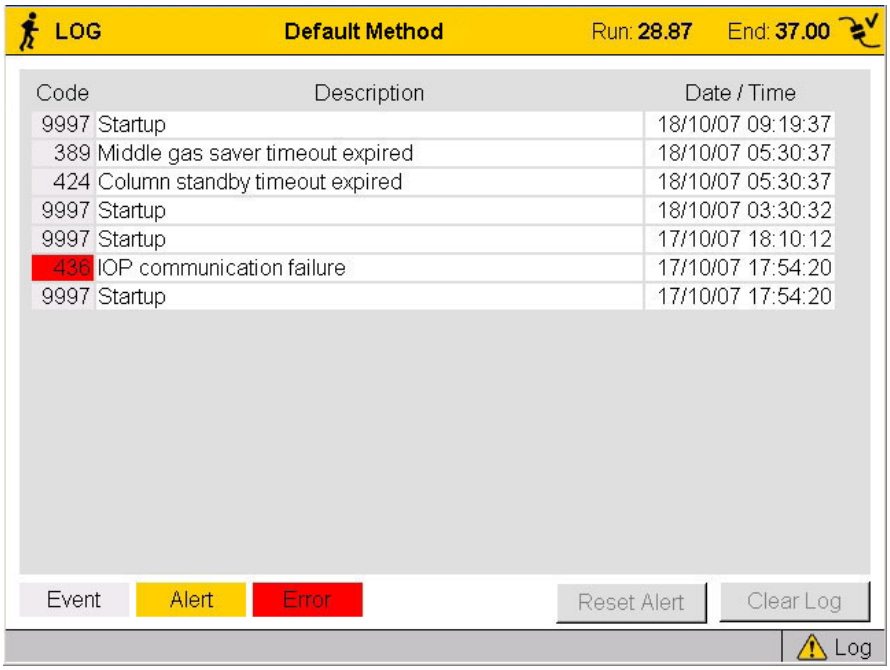


An Error[a] will be visible by a red error led.

Also in the right lower corner of the display a message Log will be visible, indicating that the Log screen has been updated with Log (Alert or Error) related information.

In case of an Event no Log will be visible.

Just touch the Log to open the log screen.



[a] A list of all errors with error conditions, can be found in Appendix A: Error Codes on page 479

There are 3 log-stats present:

Event

Will give information about all “none” error events (like Startup date time).

Alert

A fault that prevents correct function of the instrument (FID flameout or Vial missing and more). There is no dangerous situation and only a local problem in a control/module.

Actions taken by the GC:

- Fault will be reported in the error-log in yellow.
- The red error led will blink.
- The ready led will turn off (if on).
- The not ready led will turn on.
- The GC ready-out contact of the GC will switch off,
- The Main-Display will display “Not Ready”
- The module/control causing the problem will be deactivated.
- If a run was already started, it will not be interrupted.
- The AutoSampler will not inject, GC is in Not Ready state.

Error

This error is potentially dangerous for user and/or instrument. The GC will go to a safe situation for all hardware components installed. The problem can only be solved by intervention by the service engineer. After solving the error only power off/on can resolve the GC.

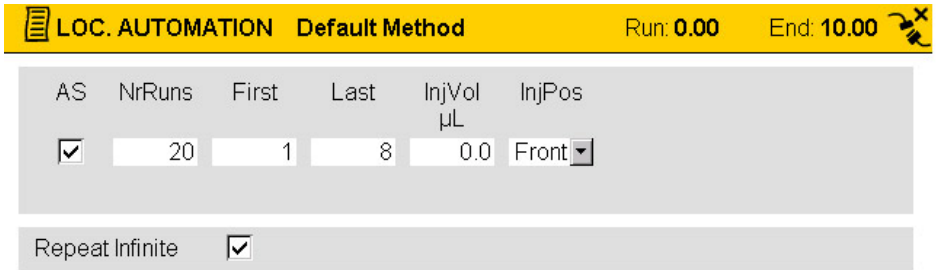
Actions taken by the GC:

- Fault will be reported in the error-log in red.
- The red error led will turn on.
- The ready led will turn off (if on).
- The not ready led will turn on.
- The ready relay of the GC will switch off.
- Switching off all heaters and Oven fan motor.
- The Main-Display will display “Error”
- The module/control causing the problem will be deactivated.
- All valves (external events) are set to default state identical to when the GC is not powered.
- All Detector electronics will be turn off.
- All Injector/detector EFC units controlling Hydrogen gas will be turned off (flow setting zero).
- If a run was already started, it will immediately aborted.
- Instrument control will be “frozen”

LOCAL AUTOMATION

Local Automation is a tool to operate multiple analysis and single method directly via the GC. The input is done via the Display and Keyboard and is only accessible when the GC is not connected to a Workstation.

To access the Automation screen, touch the local automation button in the Operation screen



The Sampler (if present) will be recognized. It is optional to disable the Sampler.

On order to loop the sample list the repeat infinite must be checked.

To start the local automation press the start button.
The run will start as soon as the 450-GC state is ready.

A local automation running button will appear. Pressing the pause button will pause the local automation run .

COMMUNICATIONS

Introduction

The 450-GC uses Ethernet® as the method of communications for instrument control and for sending digitized data to an external data system. In addition, the 450-GC is equipped with (optional) analog output ports which allow analog data to be sent to external integrators or other analog data systems.
The 450-GC is equipped to handle digital and analog data simultaneously. The Ethernet communication option allows Workstation control of the 450-GC and transmits up to three channels of detector data to the Workstation.
The (optional) analog connection can be configured with up to three detector analog outputs and 8 external timed events.
Various cable options are available for sending analog data to integrators and data.

Ethernet Communications

Ethernet refers to the type of hardware connection used to transfer digital data between the 450-GC and the data system to which it is attached.
The 450-GC has a RJ45 connector located on the rear of the GC. The Ethernet cable connects the 450-GC to the Workstation or switch on a company network.

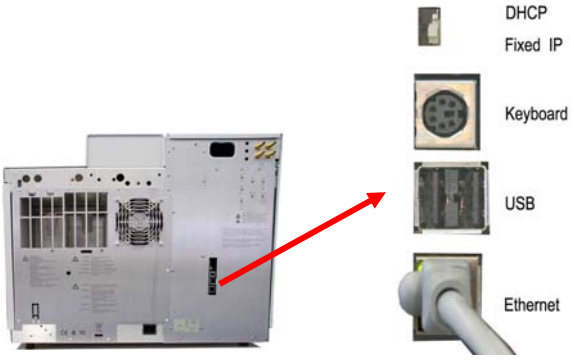
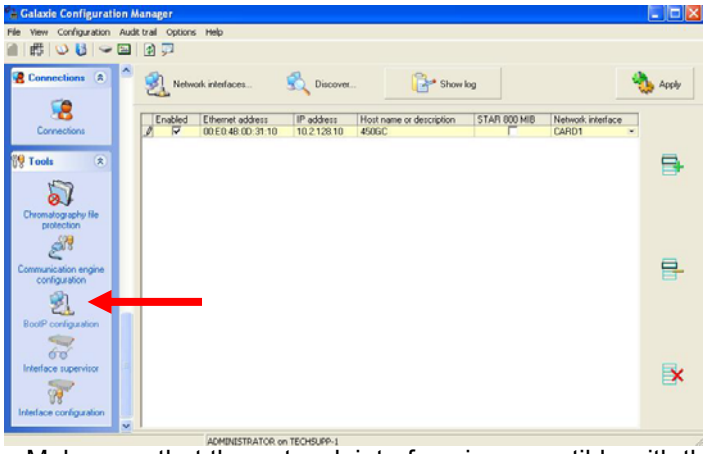


Figure 12: 450-GC Back-panel Ethernet Connection

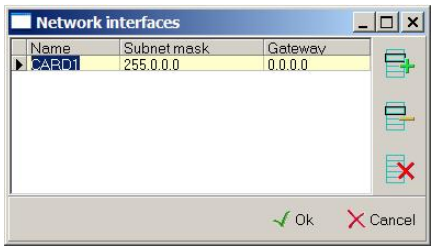
How to set IP address in Galaxie Workstation using BOOTP (DHCP)

This procedure describes how to set the IP address in Galaxie Workstation.

1. Select the network card of the computer and write down the IP-address, SubNet mask and Gateway.
2. Example IP address network card: 10.1.128.1 or can be determine by the customer.
3. Subnet mask and gateway can be determine by the customer, if not change then change the subnet mask to a value that is compatible with the instrument used.
4. Open the Galaxie configuration manager and select in “Tools” the BOOTP configuration.



5. Make sure that the network interface is compatible with the GC settings.



6. Select “Discover….” .

7. Check if the BOOTP (DHCP) switch at the rear of the GC on page 42 is in the “upper” position, switch the GC Off/On or On.

8. When the MAC address (from the GC) appears select the address and press Validate.

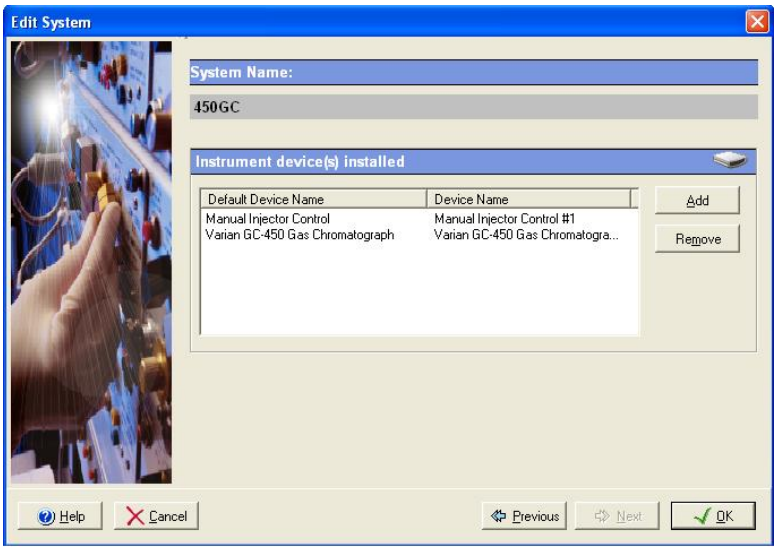
9. Fill in IP-address and the host name or description and press "**APPLY**".

10. Select "System" in the organization bar to create a new system.

11. Create a new Instrument and select the Acquisition and Sequence server and press "Next".

12. Select Groups/Project and press "Next".

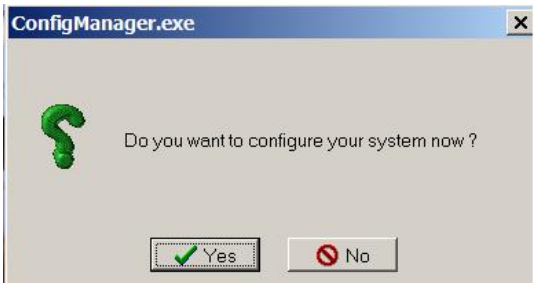
13. Add the devices (example 450-GC and **NO** sampler).



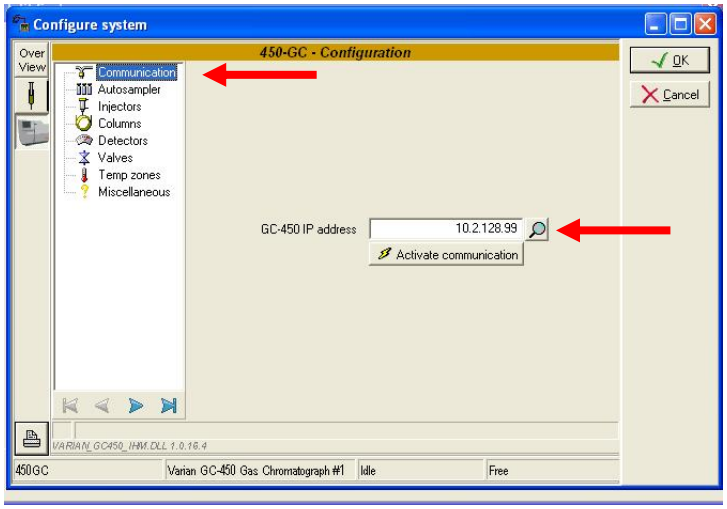
14. Only the 450-GC driver must be installed when a sampler is installed.

15. Press “OK”.

16. Press Yes to access the system.



17. Select communication and press the magnifying glass to see the instrument or type in the IP-address.



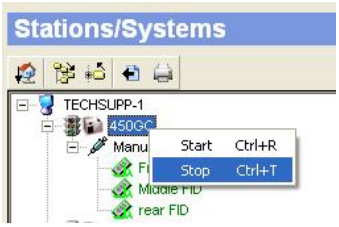
18. Press “activate communication”.

19. The next message should appear:
    a. Parameters activated.
    b. Setup received from Instrument.

20. Press OK.

21. Stop and Start the instrument.

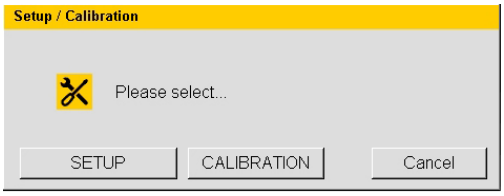
22. Instrument is ready to use with Galaxie.



How to set a Fixed IP address in the 450-GC

1. Check if the BOOTP (DHCP) switch at the rear of the GC on page 42 is in the “lower Fixed IP position”. If no, switch the GC Off/On to On.

2. Press the SETUP key and select: Setup on page 65.



3. Please continue at tab “System” on page 79.

ANALOG CABLES

The 450-GC has a number of analog cables options, depending on the device to which the cable is connected. All cables have a 15-pin Dsub connector on one end to attach to J401 (analog out) on page 36 on the 450-GC and have appropriate connectors on the other end of the cable for the devices to which they are being attached.

Partnumber	***Description***	***Picture***
392567501	Analog cable W/Sync Spade lugs. This cable is equipped with three sets of shielded analog signal pairs, terminated with spade lugs and 2 sets of sync signals, a “Sync-In” to the 450 and a “start-pot” from the 450.	
392567201	Analog out cable to old ADC board.	
392567301	Sample intro device cable (Archon/Tekmar).	
390793805	Analog cable to MIB 800 Box.	
CBLV3800	Analog cable to PAL.	

QU040.0115	Analog cable to QUMA.	
CP742629	Analog cable to QUMA/CP8400.	

Table 23: Analog Cables

SAMPLE INTRODUCTION

Sample introduction devices refer to the hardware used to introduce the sample into the Gas Chromatograph. These include AutoSamplers, liquid Injectors, gas sampling valves and liquid sampling valves. This screen also covers the pneumatic systems associated with the various sample introduction systems. The various pneumatic systems are described with the individual sample introduction systems.

AUTOSAMPLERS

Two Varian samplers are available for the 450-GC, the CP-8400 AutoSampler and the CP-8410 AutoInjector, on page 103. Also a Combi PAL sampler (Liquid, Headspace, SPME Injection System) is available for the 450-GC, more details can be found at www.varianinc.com.

LIQUID INJECTORS

There are 5 liquid Injectors available on the 450-GC Gas Chromatograph:

1. The **1177 Split/Splitless Capillary Injector on page 153** is optimized especially for split and splitless injections. The Injector is designed to accept a variety of inserts allowing tailored configurations for either the Split or Splitless injection method.

2. The **1079 PTV Injector on page 175** can be used in a variety of sampling modes including isothermal split and splitless, temperature ramped splitless, on-column and large volume injection. The 1079 PTV can be temperature programmed and cooled by cryogenics.

3. The **1041 On-Column Injector on page 205** is designed for use with wide bore capillary columns or packed columns. The standard Injector is supplied with an adapter for wide bore capillary columns. Optional adapter kits are available for 1/8" and 1/4" packed columns.

4. The **1061 Flash Vaporization Injector on page 219** is also designed for use with wide bore capillary or packed columns. This Injector uses a glass insert inside the Injector body. The standard Injector is supplied with an insert for use with wide bore capillary columns. Optional adapters are available for 1/8" and 1/4" packed columns.

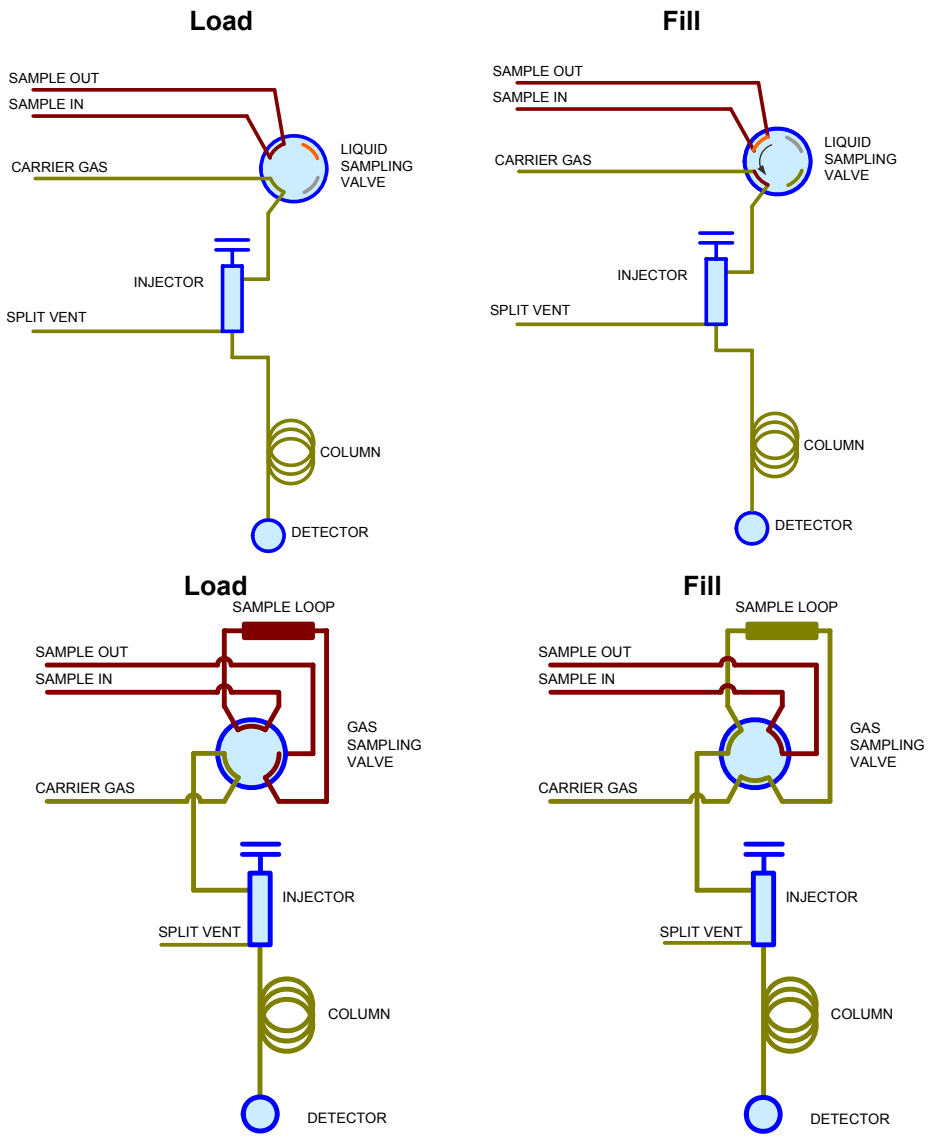
5. **The 1093 Septum-Equipped Programmable Injector (SPI), on page 231** gives optimum performance for non-vaporizing (cold) injection into conventional

fused silica capillary columns. It incorporates several unique design features to produce improved peak resolution, quantitation and versatility over other cold on-column Injectors. The result is not only a capillary on-column injection system ideally suited for analyses that cannot be done with conventional split/splitless injection, but one that gives highly accurate and precise results with a wide variety of trace to semi-trace level samples.

6. **The SPT Injector**, **on page 251** is a concentrating system for the determination of trace level volatile organic components in air and other gaseous matrices. Integrated into the top of the GC, its design provides the following features:
    - Fast heating rates.
    - Very low cryogenic consumption using liquid carbon dioxide (LCO_2) or liquid nitrogen (LN_2) cryogenic cooling.
    - Short and long trap length options.
    - Fully integrated in the GC method.

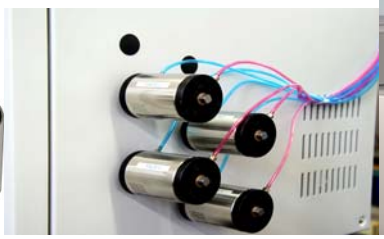
SAMPLING VALVES

The 450-GC can be configured with a wide variety of gas and liquid sampling valves. These sampling valves can be unheated or installed in a (valve) oven. Typically a sampling valve is installed in series with a liquid Injector and thus shares the pneumatic system of the Injector, but valves can also be installed with their own pneumatic systems. Optional actuators are available to automate the function on the sampling valves. In addition to sampling valves, Varian also supplies a large number of switching valve configurations.



The following are the typical locations for valves on the 450-GC:

- Unheated Valves located on the top panel of the instrument, beneath the Injector cover. The area behind the Injectors can accommodate multiple (3-possible) unheated valves.
- Dual valve ovens which replace 1 or 2 Injectors positions on the 450-GC. The single valve oven can accommodate 1 valve and an optional 1041 or 1061 Injector. The dual valve oven accommodates 1 or 2 valves.
- A multi-position valve oven which can accommodate up to 6 standard valves with 4 - 10 ports, or one 12 port or larger valve (including a Stream Selector Valve) and 4 standard valves. This valve oven is mounted over the rear Injector position and utilizes 1 heated zone.
- Up to three valves can be installed on the right side of the column oven. This is the least preferred location due to temperature cycling of the column oven and will effect the valve live negatively.



Heated Zones for Valves

If valves are located in a valve oven then they are configured in 450-GC as front, middle, rear, valve ovens. The valve oven temperature is programmed in the Oven tab . Note that when multiple valves are installed in one valve oven the temperature of the oven should always be set lower than the lowest temperature rated valve. In general valves have upper temperature ratings of 220 °C or 350 °C.

External Sample Introduction Systems

In addition to liquid Injectors and sampling valves, several external sampling systems can be used with the 450-GC. The most common are besides the CP-8400/8410 AutoSampler a Purge and Trap System and the Heated Headspace System. These sampling systems are supplied with individual operator's manuals.

Pneumatic Systems

Most sample introduction systems may be equipped with manual pneumatics or Electronic Flow Control (EFC). The following table summarizes the available pneumatics systems for liquid Injectors, sampling valves, purge and trap and heated headspace systems.

Sample Introduction System	***EFC Module***	***Optional Manual Pneumatics System***
1177 Liquid Injector	EFC21	Manual pneumatics - inlet flow controller/back pressure regulator with pressure gauge.
1079 PTV Liquid Injector	EFC21	Manual pneumatics - inlet flow controller/back pressure regulator with pressure gauge.
1041 Liquid Injector	EFC23	Manual digital flow controller with pressure gauge.
1041 Liquid Injector	EFC24	Manual pressure regulator with gauge.
1061 Liquid Injector	EFC23	Manual digital flow controller with pressure gauge.
1061 Liquid Injector	EFC24	Manual pressure regulator with gauge.
Gas or Liquid Sampling Valves	EFC24	Manual pressure regulator with gauge.
Purge and Trap with either 1177 or 1079 PTV	EFC25	Manual pneumatics - inlet flow controller/backpressure regulator with pressure gauge.
Headspace with either 1177 or 1079 PTV	EFC25	Manual pneumatics - inlet flow controller/back pressure regulator with pressure gauge.
1093 Liquid Injector (SPI)	EFC23	Manual digital flow controller with pressure gauge.

Figure 13: Pneumatic Systems for Liquid Injectors and Sampling Valves

CP-8400/8410 AUTOSAMPLER

INTRODUCTION

We strongly recommend that you read this section before you start using your CP-8400 AutoSampler or CP-8410 AutoInjector.

The CP-8400 is a 100 vial capacity AutoSampler.
The CP-8410 is an AutoInjector based on the CP-8400; it has a manually positioned tray and can accommodate up to 10, 6 and 5 each of 2 mL, 5 mL and 10 mL vials respectively.

Both the AutoSampler and AutoInjector can inject into 2 Injectors; the Sampler can inject into either one of the 2 Injectors or into both (either from the same sample vial or from adjacent vials) during the same analysis.

Both the CP-8400 and CP-8410 are preprogrammed with several injection modes (Standard On-Column, Standard Split/Splitless, Viscous, Volatile and Neat) that are optimized for most common types of samples. The User Defined mode allows access to all parameters and should be used with discretion since injection accuracy and precision may be adversely affected by incorrect choice of certain parameters. The SPME mode requires different hardware (optional). If the SPME mode is selected the other injection modes will not be available.

This section will cover AutoSampler installation, software setup and calibration to the correct sampling and injection positions, a description of the operating parameters and some examples of how you may wish to use the CP-8400/8410 to accomplish your analyses. If, after reading this section you still have questions or encounter a problem that is not covered, please contact your local Varian Technical Support for further assistance.

INSTALLING THE CP-8400/8410

If the Sampler has been installed by a service engineer, you can skip this chapter and goto page: 122.

WARNING

Before installing the Sampler, turn off the oven and any heated zones and let them cool down. Turn off any detector gases at their supply. Turn off the main power switch and unplug the power cord.

CAUTION

Use the correct (included) screws (metric) to mount the sampler.

1. Before installing the CP-8400 or CP-8410 AutoSampler, make certain your 450-GC is equipped with the appropriate Top-Cover. This cover will have a long rectangular opening for the installation of the Injector locator plate, markings indicating the front and rear installation positions for the AutoSampler and a small rectangular opening to route the CP-8400/8410 power/communication cable through.

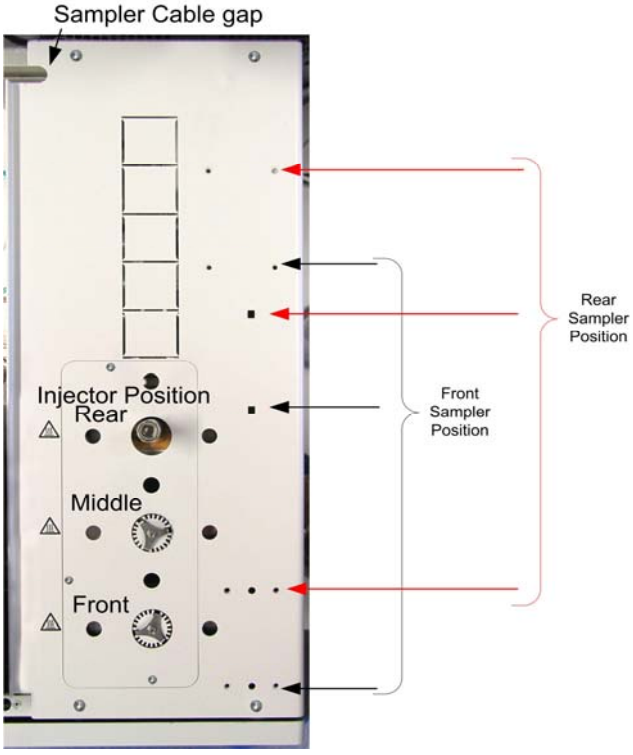


Figure 14: Top-Cover for Sampler

There are 2 positions on the 450-GC in which the CP-8400/8410 can be installed: front and rear.

- The front position allows the CP-8400/8410 to use the front and middle Injectors.
- The rear position allows the CP-8400/8410 to use the middle and rear Injectors.

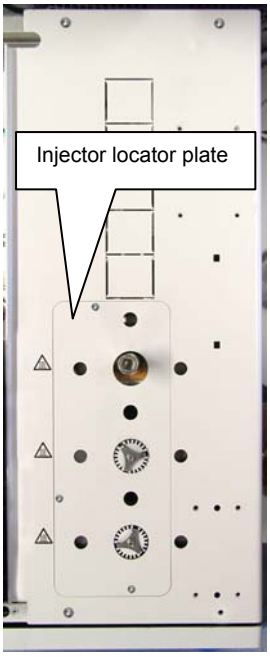
2. Remove any old equipment or old autosamplers from the top of the GC.
3. Place the CP-8400/8410 near the GC, manually rotate the Injector tower so that it does not obstruct the Sample-carrousel/tray.
4. Remove the Sample-carrousel/tray.
5. Remove the old 450-GC Top-Cover by lifting up and removing the Detector cover. Remove the Detector cover by lifting the rear of the cover and sliding it towards the rear of the GC and up. Remove the 4 screws holding the Injector cover down on the top of the GC. Retain the screws and set the old Top-Cover aside.
6. Install the sampler Top-Cover so that the guide-pins on the cover fit into the corresponding slots on the chassis. Screw the Sampler Top-Cover in place.

Remove before installing the Sampler, the screw positioned at the front-right of the Top-Cover. See Figure 14.

7. Route the CP-8400/8410 power/communication cable across the top of the GC to the left side of the GC. Plug the power cable into Aux 1 (J46) or Aux 2 (J45), on page 38 and the 25-pin D-shell into the S.I.D. 2 (J4), on page 35 connector.

8. Route the CP-8400/8410 power/communication cable through the Sample top-cover gap.

9. Install the Injector locator plate by gently pushing the circular holes over the Injector(s). Do not use excessive force. If the Injector(s) do not fit conveniently, loosen the screws holding the Injector(s) to the 450-GC chassis and adjust the Injector(s) positions so that they conveniently fit into the locator plate. The top cover has small access holes to allow you to loosen the retaining screws on the Injectors. You may need to unscrew the Injector nut and remove the inject switch to have unimpeded access to the Injector retaining screws; replace the Injector nut (if removed) so that you have proper alignment when the locator plate is installed. Once the Injector(s) are properly aligned, screw down the alignment plate and then screw down the Injector retaining screws. Remove the locator plate, replace the inject switch on each of the Injector(s) and reinstall the locator plate.



10. The CP-8400/8410 is aligned to the Top-Cover of the 450-GC by a 2 pins protruding from the bottom of the AutoSampler/AutoInjector base. These 2 pins will fit into holes on the Top-Cover.

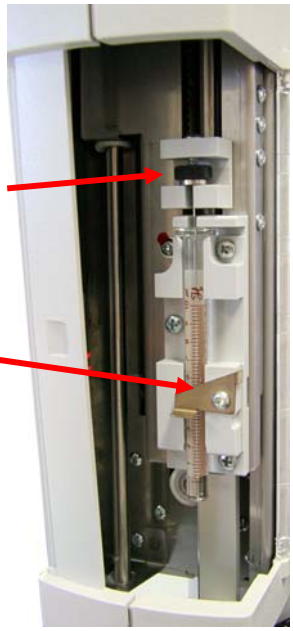
11. Place the CP-8400/8410 onto the Top-cover, aligning it with either the front or rear mounting position (See Figure 14) as appropriate.

12. The base is secured to the Top-Cover via 2 (short) screws at the rear and 3 (long) screws underneath the AutoSampler/AutoInjector sample-carrousel/tray.

13. Plug the connector of the power/communication cable into the CP-8400/8410.

14. Remove protective shipping materials from the Injector carriage and install the syringe into the AutoSampler Injector tower by opening the clear plastic door, carefully inserting the syringe into its holder and the needle through its guide, then fasten the plunger screw on the top and snap the retaining clip over the syringe.

15. Close the clear plastic sampler door.



16. Replace the left side top cover.

17. Place Waste and Solvent vial (see here for large Solvent / Wash on page 111 vial).

18. Place a capped vial into the Vial 0 location if you have a CP-8400 or the Vial 1 location if you have a CP-8410.

19. Place the CP-8400 sample-carrousel onto the base or place the CP-8410 sample tray oriented so that Vial 1 is under the arc traced by the tower.

CAUTION

If you re-locate the CP-8400/8410 to the rear position, you MUST recalibrate the Injector positions. You will not be automatically prompted to recalibrate the Injector positions. If you fail to re-calibrate the Injector positions, the AutoSampler may miss the Injector, resulting in bent syringe needles.

SAMPLER FIRST START UP

This chapter describes the GC and Sampler first time combined startup and helps you to get the system ready for use.

1. Before GC power-up it's very important to read the Warnings and Notes below!

It is important that the CP-8400/8410 is switched on prior to power-up the GC. A flashing green LED at the rear of the AutoSampler is indicating that there is communication between 450-GC and the Sampler.
Do not turn off the Sampler power switch during normal operations.

WARNING

Syringe moves automatically during operation of instrument. Keep your hands and fingers out of the injection area. Failure to do so may result in accidental needle prick/scratch.

WARNING:
BURN HAZARD

The Injector nut and locator plate may be very hot during instrument operation and should not be touched with unprotected hand.

Using SPME mode sample introduction, it is mandatory to perform the calibration process with the liquid sampling syringe provided standard with your AutoSampler.

2. Set the Sampler switch **ON**.

3. Switch the GC **ON**.

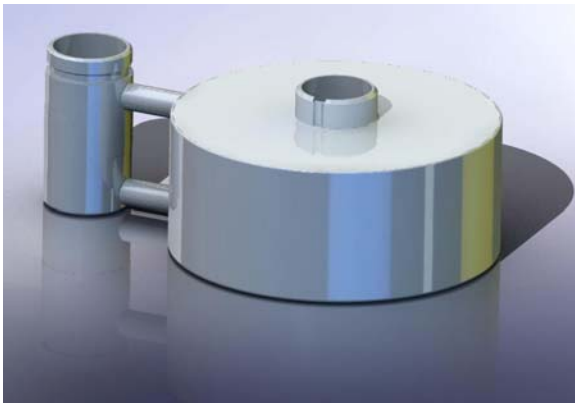
4. The GC will start initialization the system, during this process the sampler will start moving the CP-8400 carrousel, syringe and tower to check if all sensors and positions are correct. If a sample-error occurs the initialization will stop, the error will be visible in the error log. GC Power Off/On restarts the initialization process.

5. The GC/Sampler combination must be calibrated now, proceed with the next calibration chapter.

LARGE SOLVENT / WASH VIAL

Large volume Liquid Injections of 50 µL and more are become increasingly important for trace analysis. Large volume injections require an extra capacity to wash the syringe. The 5 mL wash vial does not contain sufficient solvent to run a 100-vial tray in an automated way. With the large wash vial the capacity is increasing by a factor of 60.

The Large Wash Vial is a 120 mL stainless steel vial and has 2 openings. Both use a snap cap. The vial replaces the 5 mL solvent vial.
The Large solvent vial can easily be refilled. Optional a second large solvent vial can be installed.
The vial is suitable for non-aggressive solvents. The stainless steel design makes it unbreakable and re-usable.
The large solvent vial is available as a field installation kit only (CP741158).
Additional large solvent vials can be ordered separately (CP741161).



CALIBRATION

Sampler calibration will align the Sampler components (Tower, Vials and Needle) and the GC Injector ports.
Calibration procedure is important for a trouble free working GC/Sampler combination.
Calibration must be carried out in the following cases:

- First Installation of the Sampler.
- Sampler has been removed from the GC and after maintenance/repair back on the GC.
- Bending Sample needles.
- Other Injector positions used (back or front).
- If the Injector has been removed calibration is mandatory.

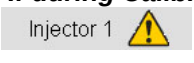
Do not recalibrate the Injector positions after normal Injector maintenance such as changing septa or inserts.

Two calibration methods are present:

- Vial calibration, on page 113.

- GC Injector calibration, on page 116.

If during Calibration a question mark appears in a yellow triangle,



this will indicate that the calibration is not correct!

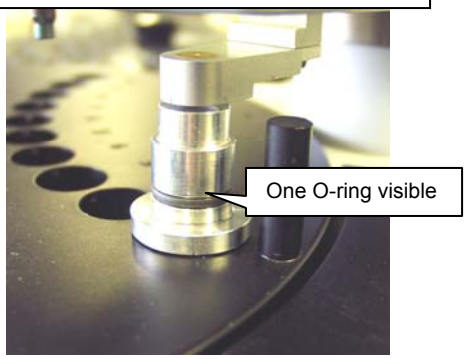
Vial calibration

The Vial Calibration procedure will allow the AutoSampler/AutoInjector to learn the tower and carrousel/tray positions so that the syringe is positioned directly over the middle of the first vial in the sample-carrousel (Vial 0) or the middle of the first 2 mL vial of the AutoInjector sample tray (Vial 1).
The CP-8400 may use a number of different sample vial form factors; however, you should use all of the same vials for each session.
Mixing different vials is strongly discouraged. If you calibrate using one vial type and then begin using another vial type with a different height, it is strongly recommended that you recalibrate the Vial 0 Position to avoid bent needles and other minor problems.

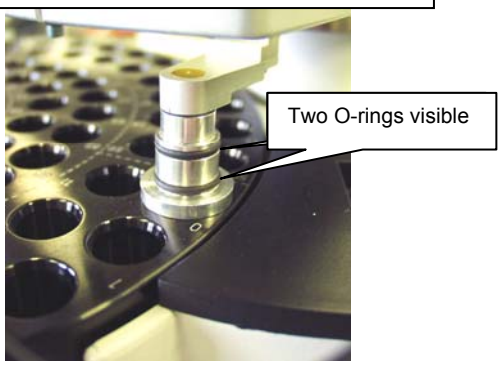
In order to accurately calibrate Vial positions use the calibration tool (P/N: CP740889). The calibration tool simulates a standard vial. The tool is used for both the CP-8400 and CP-8410 AutoSamplers.

The calibration tool can be placed (depending of the carrousel/tray-material) in 2 different ways in the tray.

CP-8410 (polymer tray) calibration tool in Vial position 1

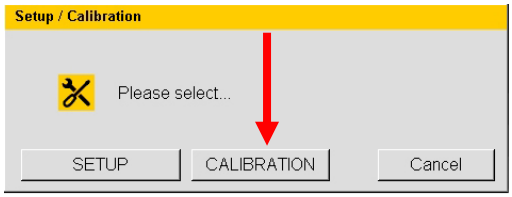


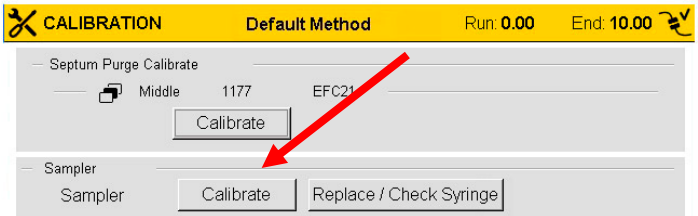
CP-8400 (metal carrousel) calibration tool in Vial position 0



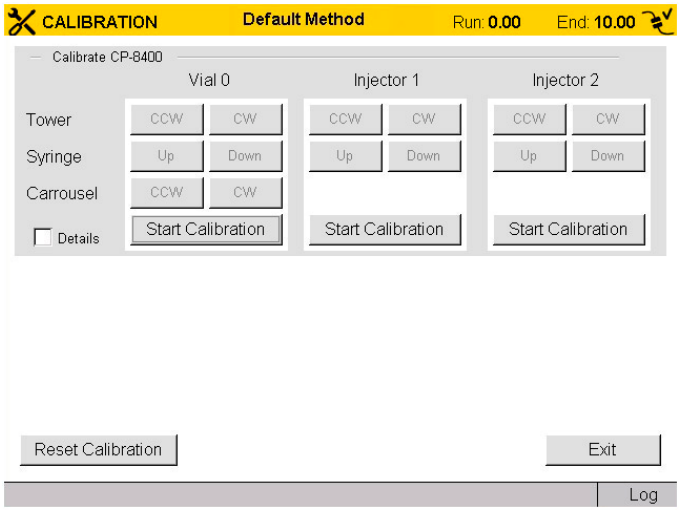
To assure consistent operation, always approach the final Tower and Carrousel calibration position with CW (clockwise) movements. To help facilitate this rule, the carrousel will backup 10 steps when a CCW (counterclockwise) action is selected but will move 1 step per move action when a CW (clockwise) carrousel rotation is selected.

1. Place the Calibration tool in Vial 0 or Vial 1
2. Press the SETUP key and select CALIBRATION, select on the next page Sampler CALIBRATE.





3. The calibration screen will be visible.



Calibrating the CP-8400 Vial 0 position involves manipulating the positions of 3 different components (Tower, Syringe and Carrousel).
Calibrating the CP-8410 Vial 1 position involves manipulating the positions of 2 different components (Tower and Syringe).

4. Press “Start Calibration”. The CP-8400/8410 will rotate the tower, rotate the sample-carrousel (not for the CP-8410) and lower the syringe sled to the approximate location of Vial 0 (or Vial 1 if CP-8410). After the

CP-8400/8410 has found the approximate Vial 0 (or Vial 1) position, visually check that the syringe sled is not resting on the Vial top.
If the syringe sled is resting on the vial top, press the Syringe “Up” button until the syringe sled is clear of the Vial top by approximately 1 mm.

5. Start the calibration process by adjusting the tower position. Move the Tower CCW or CW until the needle-guide is exactly cover the top of the calibration tool.

6. For CP-8400, set the sample-carrousel position. You may have to switch back and forth between the tower and sample-carrousel one or more times to determine the optimum rotational positions.

7. Adjust the syringe sled height so that the needle is just barely above the calibration tool top. You should be able to see about a paper’s thickness of space between the syringe sled needle guide and the top of the calibration tool. The penetration depth into the vial is measured from the bottom of the needle guide, so this height will give you optimum penetration depth.

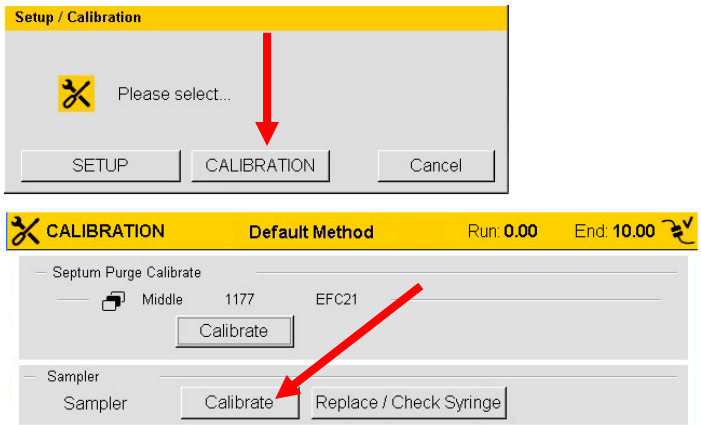
8. Press the “Accept” button if the calibration of Vial 0 or 1 is complete, the calibration data will be saved.

9. Press “Exit” button to exit the calibration screen, without saving the calibration data.

If the used Vial is shorter/longer then the calibration tool it’s advisable to repeat <u>only</u> the Syringe calibration.

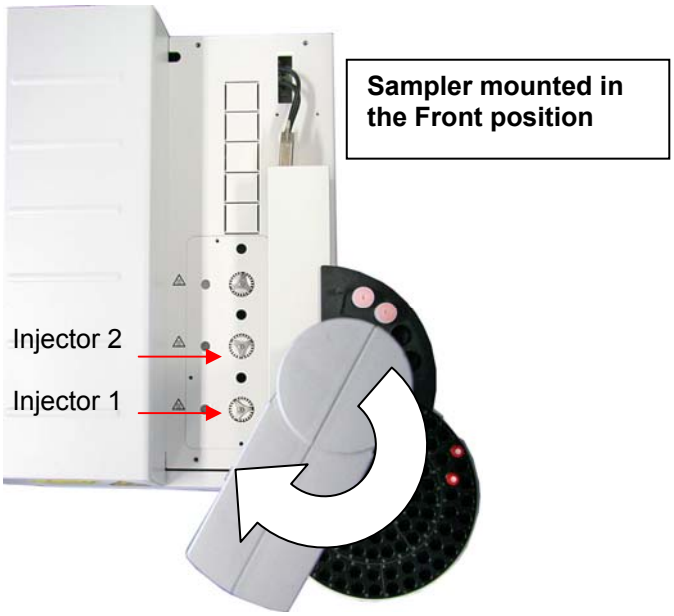
GC Injector calibration

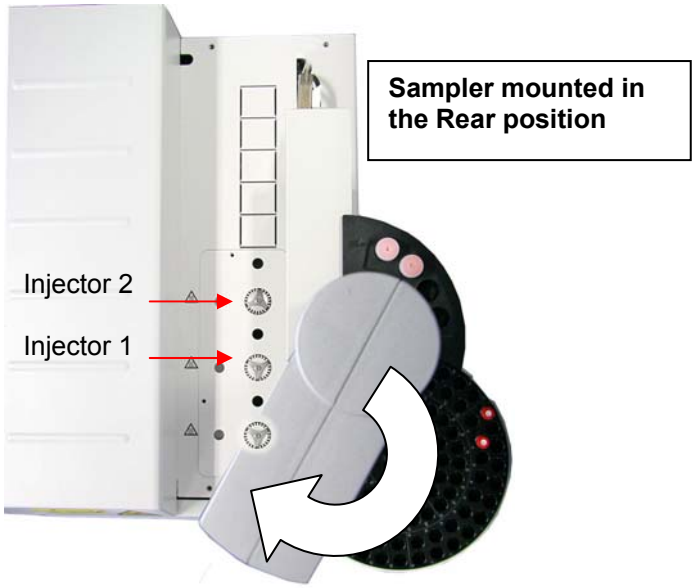
1. Press the SETUP key and select CALIBRATION, select on the next page Sampler CALIBRATE.

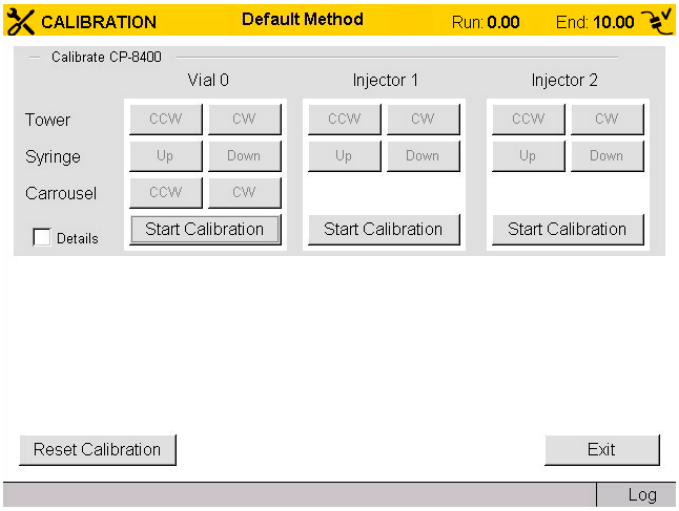


2. The calibration screen will be visible.

3. Before starting the calibration identify Injector 1 and Injector 2. Injector 1 is the first Injector the Tower will encounter when it rotates clockwise to reach the Injector(s). The second Injector is Injector 2. Please notice the difference when the sampler is mounted in the Front or Rear (next page) position.







WARNING

Syringe moves automatically during operation of instrument. Keep your hands and fingers out of the injection area. Failure to do so may result in accidental needle prick/scratch.

4. On the calibration screen Injector 1 press “Start Calibration” button. The Injector Tower rotates so that it is over Injector 1 position and lowers the syringe sledge so that it is over the Injector.

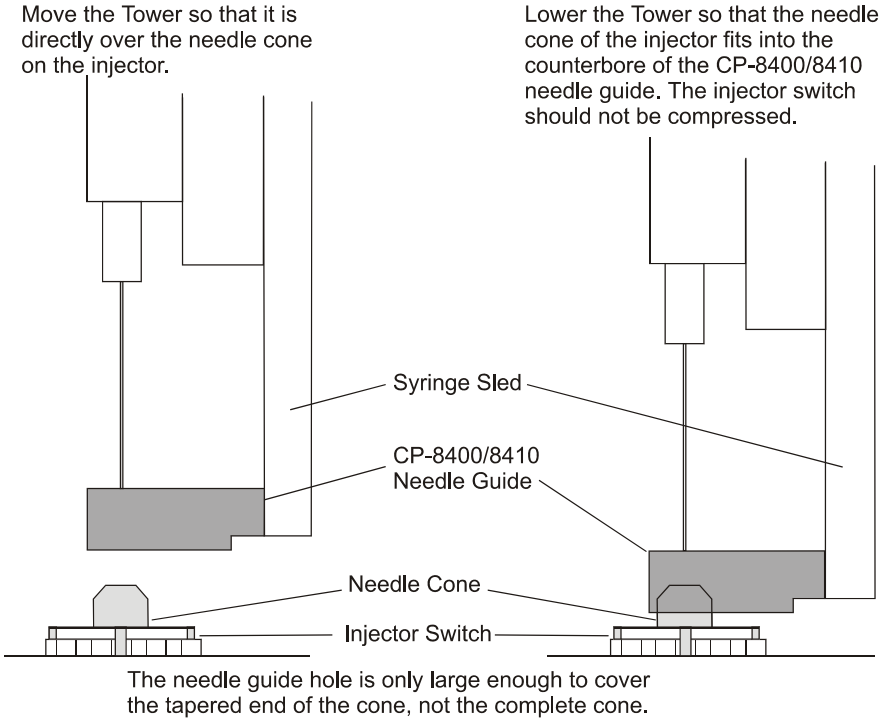
5. If the needle guide descends so that it is either resting on the inject switch or below the needle cone on the inject switch, use the “Up” button move the sled up (10 steps) until the needle guide is just above the Injector cone.

6. Using the “CCW” (counterclockwise) and “CW” (clockwise) button. Position the tower directly over the Injector Inject Switch Locator cone.

7. Adjust the height (using the “Up” and “Down” button) of the needle guide such that the sled is just resting on or very slightly depressing the Injector Switch and is covering the locator cone.

8. Press the “Accept” button if the calibration of Injector 1 is complete, the calibration data will be saved.

9. Calibrate Injector 2 (if present) using the above mentioned procedure.



10. Press the “Exit” button if calibration is finished or leaving the calibration page without saving is needed.

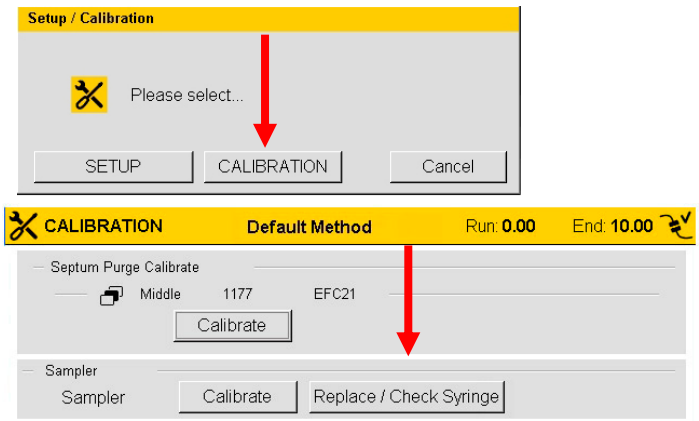
The “Reset Calibration” button will restore the factory settings to all calibration values.

SYRINGE CHECK/REPLACE

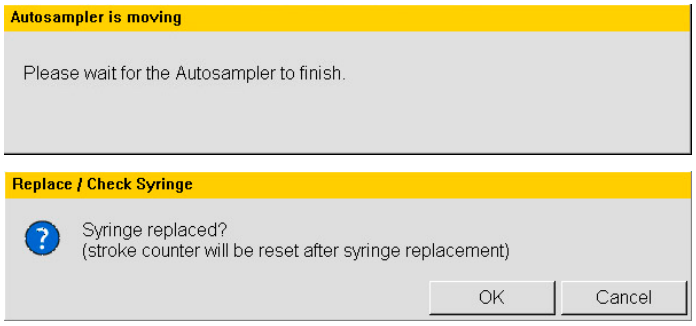
For changing the syringe ***size*** continue on the next page.

If the syringe has to be replaced by the same size follow the procedure below:

1. Press the SETUP key and select CALIBRATION, select on the next page Sampler Change Syringe.



2. The next massage will pop-up and the Sampler Tower will go to his Syringe Replace/Check Syringe position.



3. Check or Replace the Syringe when the above pop-up message appears.

4. After syringe replacement the syringe plunger stroke counter can be set to zero, press OK.

5. After Syringe check (no reset of the counter necessary), press Cancel.

SYRINGE SIZE CHANGE

For changing the syringe size the next procedure MUST be followed:

1. Change syringe size in Setup tab “SampleDelivery”.
2. Press OK to leave Setup.
3. Follow the Syringe Check/Replace procedure (previous page).

Follow the correct working order!

OPERATION

PARAMETERS AND QUICK REFERENCE

The layout of the screens and the placement of the parameters followed the philosophy of keeping those parameters that an operator may wish to change for a series of samples (such as the injection volume, vial range, GC method) within the Automation pages for quick access and change.

Those parameters that drive the AutoSampler actions such as clean, viscosity settings and needle residence times are located within the AutoSampler Method.

The CP-8400/8410 AutoSampler is preprogrammed with settings and actions that are appropriate for most common analyses. The following diagrams represent the hierarchy of screens that follow from selecting the indicated key on the 450-GC front panel.

SAMPLER CONFIGURATION

The AutoSampler will be delivered in one of two (Liquid or SPME) configurations.

Default is the Liquid Sampling configuration.

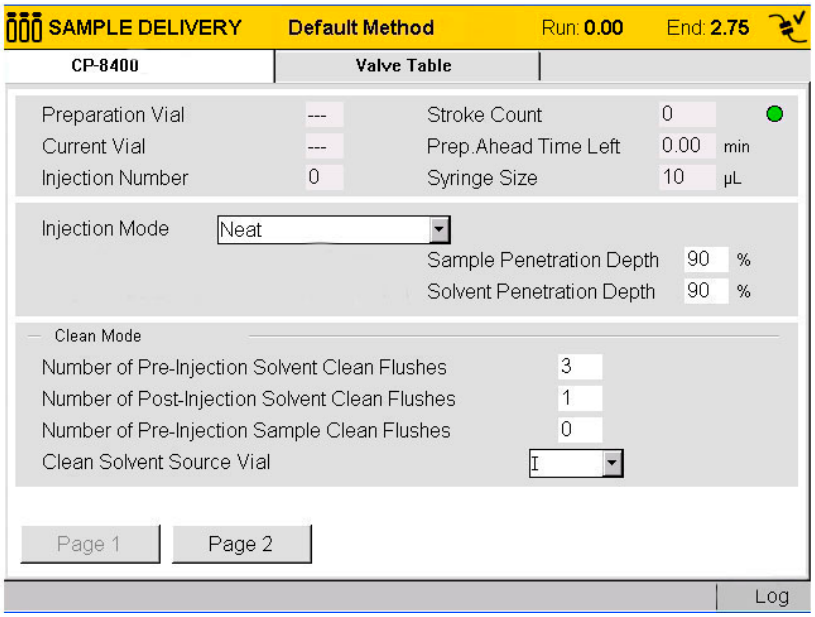
The SPME operation can be thought of as fundamentally reversed from liquid sampling in terms of the actions the plunger performs during the sampling process. In liquid sampling, the plunger is at its home state more or less fully descended and sampling is done by withdrawing the plunger from the syringe barrel in order to take an aliquot for injection. In SPME however, the plunger is at the top of its travel when it is in the home position and the SPME fiber must be exposed by inserting the plunger into the fiber holder barrel to expose the fiber.

The SPME Sampling mode can not be selected by the 450-GC display/keyboard. In order to configure the system to use SPME you must set a switch on the syringe sled to the correct position and install the special SPME fiber holder block. This means of setting up the system to use SPME was adopted to avoid unintentional damage to either SPME hardware or syringes. Ones the installation is completed, the 450-GC will recognize the sampler mode.

- Installation of the SPME mode, on page 142.
- Installation of the Liquid Sampling mode, on page 149.

SAMPLE DELIVERY PARAMETERS

The 450-GC Sample Delivery page has parameters that represent the most frequently used parameters, however predefined modes of operation limit the need to access all parameters.



The CP-8400 (liquid) is preprogrammed with 6 injection modes designed to optimize your analysis for:

1. Standard On-Column on page 124 (Standard OC)
2. Standard Split/Splitless on page 124 (Standard Split/Splitless)
3. Neat samples on page 126 (Neat)
4. Viscous samples on page 128 (Viscous)
5. Volatile samples on page 129 (Volatile)
6. User Defined on page 131 (User Defined)

Choosing 1 of these 5 injection modes will preset critical parameters for optimum performance and limit the parameters you need to set. If you prefer to develop your own injection method for your CP-8400/8410, the 6th CP-8400/8410 operation mode user defined (User Defined), allows access to the full suite of CP-8400/8410 parameters. You should **exercise care** when using the user defined mode since choices for some parameters may have unintended consequences.

Standard On-Column and Standard Split/Splitless Sampling Modes

Internal Standard is not permitted with Standard Sampling modes.

Standard mode parameters are set for samples dissolved in common solvents such as iso-octane.

The Standard sampling mode for **On-Column** performs the following sequence of actions:

All CleanStrokes- and Flush-Volumes are limited to 75% of the syringe capacity.

Sampler sequence	Standard On-column - mode	SyringeSize (µL)						
		1	2	5	10	50	100	250
1	*If used: Pre-Injection Solvent Clean Flushes* (solvent ⟶ waste)							
	- Volume (µL)	0.75	1.5	3.75	7.5	37.5	75	187.5
	- Plunger Speed Up (µL/s)	0.5	1	2.5	5	10	20	50
	- Plunger Speed Down (µL/s)	5	10	25	50	25	50	62.5
2	*If used: Any Pre-Injection Sample Clean Flushes* (sample ⟶ waste)							
	- Volume (µL)	0.75	1.5	3.75	7.5	37.5	75	187.5
	- Plunger Speed Up (µL/s)	0.5	1	2.5	5	10	20	50
	- Plunger Speed Down (µL/s)	5	10	25	50	25	50	62.5
3	*Clean Strokes* (sample ⟶ sample)							
	- Plunger Speed Up (µL/s)	0.2	0.4	1	2	10	20	50
	- Plunger Speed Down (µL/s)	5	10	25	50	25	50	62.5
	- Volume For Fill Strokes (µL)	0.75	1.5	3.75	7.5	37.5	75	187.5
	- Number Of Clean Strokes	5	5	5	5	5	5	5
	- Viscosity Delay (s)	0.0	0.0	0.0	0.0	0.0	0.0	0.0
4	*Draw Sample*							
	- Plunger Speed During Fill (µL/s)	0.2	0.4	1	2	10	20	50
	- Viscosity Delay (s)	0.0	0.0	0.0	0.0	0.0	0.0	0.0

Sampler sequence	Standard On-column - mode	SyringeSize (µL)						
		1	2	5	10	50	100	250
5	*Air Plug After sample*							
	- Volume (µL)	0.1	0.2	0.5	0.5	0.5	2	4
6	*Inject Sample*							
	- Plunger Speed During Injection (µL/s)	0.5	1	2.5	5	5	10	12.5
	- Pre Injection Delay (s)	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	- Post Injection Delay (s)	6.0	6.0	6.0	6.0	6.0	6.0	6.0
7	*If used: Any Post Injection Solvent Clean Flushes* (solvent ⟶ waste)							
	- Volume (µL)	0.75	1.5	3.75	7.5	37.5	75	187.5
	- Plunger Speed Up (µL/s)	0.5	1	2.5	5	10	20	50
	- Plunger Speed Down (µL/s)	5	10	25	50	25	50	62.5

Table 24: Injection mode parameters for On-Column

Internal Standard is not permitted with Standard Sampling modes.

Standard mode parameters are set for samples dissolved in common solvents such as iso-octane.

The Standard sampling mode for **Split/Splitless** performs the following sequence of actions:

All CleanStrokes- and Flush-Volumes are limited to 75% of the syringe capacity.

Sampler sequence	Standard Split-/Splitless - mode	SyringeSize (µL)						
		1	2	5	10	50	100	250
1	*If used: Pre-Injection Solvent Clean Flushes* (solvent ⟶ waste)							
	- Volume (µL)	0.75	1.5	3.75	7.5	37.5	75	187.5
	- Viscosity Delay (s)	9.9	9.9	9.9	9.9	9.9	9.9	9.9
	- Plunger Speed Up (µL/s)	0.5	1	2.5	5	10	20	50
	- Plunger Speed Down (µL/s)	5	10	25	50	25	50	62.5
2	*If used: Any Pre-Injection Sample Clean Flushes* (sample ⟶ waste)							
	- Volume (µL)	0.75	1.5	3.75	7.5	37.5	75	187.5
	- Viscosity Delay (s)	9.9	9.9	9.9	9.9	9.9	9.9	9.9
	- Plunger Speed Up (µL/s)	0.5	1	2.5	5	10	20	50

Sampler sequence	Standard Split-/Splitless - mode	SyringeSize (µL)						
		1	2	5	10	50	100	250
	- Plunger Speed Down (µL/s)	5	10	25	50	25	50	62.5
3	*Clean Strokes* (sample ⟶ sample)							
	- Plunger Speed Up (µL/s)	0.2	0.4	1	2	10	20	50
	- Plunger Speed Down (µL/s)	5	10	25	50	25	50	62.5
	- Volume For Fill Strokes (µL)	0.75	1.5	3.75	7.5	37.5	75	187.5
	- Number Of Clean Strokes	5	5	5	5	5	5	5
	- Viscosity Delay (s)	0.0	0.0	0.0	0.0	0.0	0.0	0.0
4	*Draw Sample*							
	- Plunger Speed During Fill (µL/s)	0.2	0.4	1	2	10	20	50
	- Viscosity Delay (s)	0.0	0.0	0.0	0.0	0.0	0.0	0.0
5	*Air Plug After sample*							
	- Volume (µL)	0.1	0.2	0.5	0.5	0.5	2	4
6	*Inject Sample*							
	- Plunger Speed During Injection (µL/s)	5	10	25	50	25	50	62.5
	- Pre Injection Delay (s)	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	- Post Injection Delay (s)	6.0	6.0	6.0	6.0	6.0	6.0	6.0
7	*If used: Any Post Injection Solvent Clean Flushes* (solvent ⟶ waste)							
	- Volume (µL)	0.75	1.5	3.75	7.5	37.5	75	187.5
	- Plunger Speed Up (µL/s)	0.5	1	2.5	5	10	20	50
	- Plunger Speed Down (µL/s)	5	10	25	50	25	50	62.5

Table 25: Injection mode parameters for Split-/Splitless

Neat Sampling Mode

Internal Standard is not permitted with Neat Sampling mode.

Neat mode parameters are set for samples that are normally liquids that will be injected without dilution.

The Neat sampling mode performs the following sequence of actions:

All CleanStrokes- and Flush-Volumes are limited to 75% of the syringe capacity.

Sampler sequence	Neat - mode	SyringeSize (µL)						
		1	2	5	10	50	100	250
1	*If used: Pre-Injection Solvent Clean Flushes* (solvent ⟶ waste)							
	- Volume (µL)	0.75	1.5	3.75	7.5	37.5	75	187.5
	- Viscosity Delay (s)	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	- Plunger Speed Up (µL/s)	0.5	1	2.5	5	10	20	50
	- Plunger Speed Down (µL/s)	5	10	25	50	25	50	62.5
2	*If used: Any Pre-Injection Sample Clean Flushes* (sample ⟶ waste)							
	- Volume (µL)	0.75	1.5	3.75	7.5	37.5	75	187.5
	- Viscosity Delay (s)	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	- Plunger Speed Up (µL/s)	0.5	1	2.5	5	10	20	50
	- Plunger Speed Down (µL/s)	5	10	25	50	25	50	62.5
3	*Coating Strokes* (same movement as Pre-Injection Sample Clean Flushes)							
	- Volume (µL)	0.75	1.5	3.75	7.5	37.5	75	187.5
	- Viscosity Delay (s)	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	- Number of Coating Strokes	1	1	1	1	1	1	1
	- Plunger Speed Up (µL/s)	0.5	1	2.5	5	10	20	50
	- Plunger Speed Down (µL/s)	5	10	25	50	25	50	62.5
3	*Clean Strokes* (sample ⟶ sample)							
	- Plunger Speed Up (µL/s)	0.2	0.4	1	2	10	20	50
	- Viscosity Delay (s)	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	- Plunger Speed Down (µL/s)	5	10	25	50	25	50	62.5
	- Volume For Fill Strokes (µL)	1	2	5	10	50	100	250
	- Number Of Clean Strokes	3	3	3	3	3	3	3
4	*Draw Sample*							
	- Plunger Speed During Fill (µL/s)	0.2	0.4	1	2	10	20	50
	- Viscosity Delay (s)	0.0	0.0	0.0	0.0	0.0	0.0	0.0
5	*Air Plug After sample*							
	- Volume (µL)	0.1	0.2	0.5	0.5	0.5	2	4
6	*Inject Sample*							
	- Plunger Speed During Injection (µL/s)	5	10	25	50	25	50	62.5
	- Pre Injection Delay (s)	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	- Post Injection Delay (s)	0.0	0.0	0.0	0.0	0.0	0.0	0.0

Sampler sequence	Neat - mode	SyringeSize (µL)						
		1	2	5	10	50	100	250
7	*If used: Any Post Injection Solvent Clean Flushes* (solvent ⟶ waste)							
	- Volume (µL)	0.75	1.5	3.75	7.5	37.5	75	187.5
	- Plunger Speed Up (µL/s)	0.5	1	2.5	5	10	20	50
	- Plunger Speed Down (µL/s)	5	10	25	50	25	50	62.5

Table 26: Injection mode parameters for Neat

Viscous Sampling Mode

Viscous mode is designed for samples that either are viscous or are dissolved in a viscous solvent such as cyclohexanol.
Viscous mode actions are summarized below:

All CleanStrokes- and Flush-Volumes are limited to 75% of the syringe capacity.

Sampler sequence	Viscous - mode	SyringeSize (µL)						
		1	2	5	10	50	100	250
1	*If used: Pre-Injection Solvent Clean Flushes* (solvent ⟶ waste)							
	- Volume (µL)	0.75	1.5	3.75	7.5	37.5	75	187.5
	- Viscosity Delay (s)	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	- Plunger Speed Up (µL/s)	0.5	1	2.5	5	10	20	50
	- Plunger Speed Down (µL/s)	5	10	25	50	25	50	62.5
2	*If used: Any Pre-Injection Sample Clean Flushes* (sample ⟶ waste)							
	- Volume (µL)	0.75	1.5	3.75	7.5	37.5	75	187.5
	- Viscosity Delay (s)	9.9	9.9	9.9	9.9	9.9	9.9	9.9
	- Plunger Speed Up (µL/s)	0.1	0.2	0.5	1	5	10	25
	- Plunger Speed Down (µL/s)	5	10	25	50	25	50	62.5

Sampler sequence	Viscous - mode	SyringeSize (µL)						
		1	2	5	10	50	100	250
3	*If used: Internal Standard*							
	- Plunger Speed Up (µL/s)	0.5	1	2.5	5	10	20	50
	- Plunger Speed Down (µL/s)	5	10	25	50	25	50	62.5
	- Air Gap (µL) (if used)	0.1	0.2	0.5	0.5	0.5	2.0	4.0
4	*Draw Sample*							
	- Plunger Speed During Fill (µL/s)	0.1	0.2	0.5	1	5	10	25
	- Viscosity Delay (s)	9.9	9.9	9.9	9.9	9.9	9.9	9.9
5	*Air Plug After sample*							
	- Volume (µL)	0.1	0.2	0.5	0.5	0.5	2	4
6	*Inject Sample*							
	- Plunger Speed During Injection (µL/s)	0.5	1	2.5	5	10	20	50
	- Pre Injection Delay (s)	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	- Post Injection Delay (s)	12.0	12.0	12.0	12.0	12.0	12.0	12.0
7	*If used: Any Post Injection Solvent Clean Flushes* (solvent ⟶ waste)							
	- Volume (µL)	0.75	1.5	3.75	7.5	37.5	75	187.5
	- Plunger Speed Up (µL/s)	0.5	1	2.5	5	10	20	50
	- Plunger Speed Down (µL/s)	5	10	25	50	25	50	62.5

Table 27: Injection mode parameters for Viscous

Volatile Sampling Mode

This mode is designed for samples dissolved in highly volatile solvents such as pentane.

Volatile mode actions are summarized below:

All CleanStrokes- and Flush-Volumes are limited to 75% of the syringe capacity.

Sampler sequence	Volatile - mode	SyringeSize (µL)						
		1	2	5	10	50	100	250
1	*If used: Pre-Injection Solvent Clean Flushes* (solvent ⟶ waste)							
	- Volume (µL)	0.75	1.5	3.75	7.5	37.5	75	187.5
	- Viscosity Delay (s)	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	- Plunger Speed Up (µL/s)	0.5	1	2.5	5	10	20	50
	- Plunger Speed Down (µL/s)	5	10	25	50	25	50	62.5
2	*If used: Any Pre-Injection Sample Clean Flushes* (sample ⟶ waste)							
	- Volume (µL)	0.75	1.5	3.75	7.5	37.5	75	187.5
	- Viscosity Delay (s)	9.9	9.9	9.9	9.9	9.9	9.9	9.9
	- Plunger Speed Up (µL/s)	0.1	0.2	0.5	1	5	10	25
	- Plunger Speed Down (µL/s)	5	10	25	50	25	50	62.5
3	*if used: Internal Standard*							
	- Plunger Speed Up (µL/s)	0.5	1	2.5	5	10	20	50
	- Plunger Speed Down (µL/s)	5	10	25	50	25	50	62.5
	- Air Gap (µL) (if used)	0.1	0.2	0.5	0.5	0.5	2.0	4.0
4	*Draw Sample*							
	- Plunger Speed During Fill (µL/s)	0.1	0.2	0.5	1	5	10	25
	- Viscosity Delay (s)	9.9	9.9	9.9	9.9	9.9	9.9	9.9
5	*Air Plug After sample*							
	- Volume (µL)	0.1	0.2	0.5	0.5	0.5	2	4
6	*Inject Sample*							
	- Plunger Speed During Injection (µL/s)	0.5	1	2.5	5	10	20	50
	- Pre Injection Delay (s)	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	- Post Injection Delay (s)	12.0	12.0	12.0	12.0	12.0	12.0	12.0
7	*If used: Any Post Injection Solvent Clean Flushes* (solvent ⟶ waste)							
	- Volume (µL)	0.75	1.5	3.75	7.5	37.5	75	187.5
	- Plunger Speed Up (µL/s)	0.5	1	2.5	5	10	20	50
	- Plunger Speed Down (µL/s)	5	10	25	50	25	50	62.5

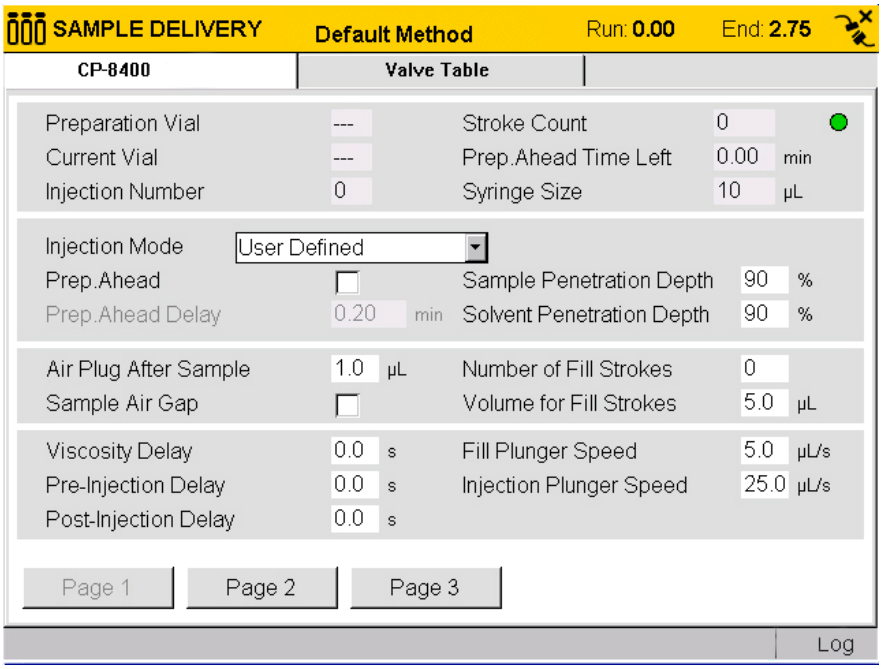
Table 28: Injection mode parameters for Volatile

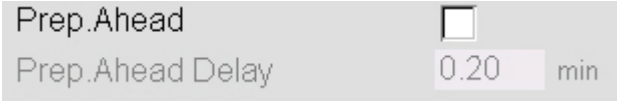
User Defined Sampling Mode

Allows access to all AutoSampler parameters.

This mode of operation should be used with caution since some parameters, if not optimally set, can produce poor area precision and anomalous results. User Defined mode has three pages of parameters.

Page 1





Prep.Ahead

This parameter is used to select whether the AutoSampler will sample for the next injection immediately after the current chromatographic run is completed or to cause the AutoSampler to sample while the current run is in progress thus improving throughput. Prep-Ahead works for either liquid or SPME sampling modes. If you set the Prep-Ahead, the system will use the Prep-Ahead Delay time entered into the method to determine when to begin sampling for the next run. The Prep-Ahead timer will begin after the AutoSampler has completed all

of its actions for the current run. For instance, if you have dual or duplicate injections selected and the second injection is set to take place 0.5 minutes into the run, the Prep-Ahead timer for the next sample will start once the second injection is finished. So if you have set the Prep-Ahead timer to 10 minutes, the system will actually start sampling at around 10.5 minutes into the run. If your sample is either heat or light sensitive you may wish to leave the Prep-Ahead blank. Note that the Prep-Ahead timer is independent of the run time; this allows you to set a next sampling time while the instrument is either equilibrating or stabilizing if the sample may be heat or light sensitive. You will have to experiment with sample and solvent flushes along with any other pre-injection settings for your AutoSampler to determine the correct Prep-Ahead time for optimum cycle time and minimum wait before injection.

Sample Penetration Depth	90	%
Solvent Penetration Depth	90	%

Sample Penetration Depth (0-100%)

This parameter defines how far the needle penetrates through the septum into the sample vial. The percentage refers to the distance from the bottom of the vial septum to the bottom of the vial interior. The valid range is settable in 1% steps. The default is 90%.

Solvent Penetration Depth (0-100%)

This parameter defines how far into the solvent vials the needle will penetrate. The percentage refers to the distance from the bottom of the vial septum to the bottom of the clean vial interior. There is only one penetration depth for all three clean/Internal Standard vials. This parameter is settable in 1% steps. The default is 90%.

Air Plug After Sample	1.0	µL	Number of Fill Strokes	0	
Sample Air Gap	☐		Volume for Fill Strokes	5.0	µL

Air Plug After Sample

This parameter defines the volume of air drawn up after the sample has been drawn into the syringe.

Sample Air Gap

Setting this parameter will cause the syringe plunger to be withdrawn an amount corresponding to 1 µL (depending of the syringe size) prior to the syringe sled descending and the sample being drawn up. Leaving this this parameter blank will cause no upper air gap to be formed. The default value is blank.

Number of Fill Strokes (0-99)

This parameter defines how many times the syringe plunger will be “pumped” (withdrawn and then advanced). This parameter is required to effectively flush the syringe with the sample to remove bubbles and prevent dilution of the sample by residual solvent from wash processes. The syringe will use the Plunger speed parameters as defined in the viscosity settings page. The default value is 0.

Fill Volume for Fill Strokes

This parameter defines the volume that will be used by the fill strokes specified above. This volume will usually be equal to or larger than the sample volume specified in the Samplelist.

Viscosity Delay	0.0	s	Fill Plunger Speed	5.0	µL/s
Pre-Injection Delay	0.0	s	Injection Plunger Speed	25.0	µL/s
Post-Injection Delay	0.0	s			

Viscosity Delay (sec)

Defines the amount of time the system will wait after the syringe plunger is withdrawn, drawing sample into the syringe. This time is required to allow samples with high viscosity to migrate into the syringe. The syringe remains in its loading position with the needle at its programmed depth in the sample vial for the duration of the Viscosity delay. The default value is 0.0.

Pre-Injection Delay (0-99.9 sec)

Specifies the length of time the needle will remain in the Injector prior to the advancing of the syringes plunger to expel its contents into the Injector. The default value is 0.0 sec.

Post-Injection Delay (0-99.9 sec)

Specifies the length of time the needle will remain in the Injector after the syringes contents have been completely expelled. The default value is 0.0 sec.

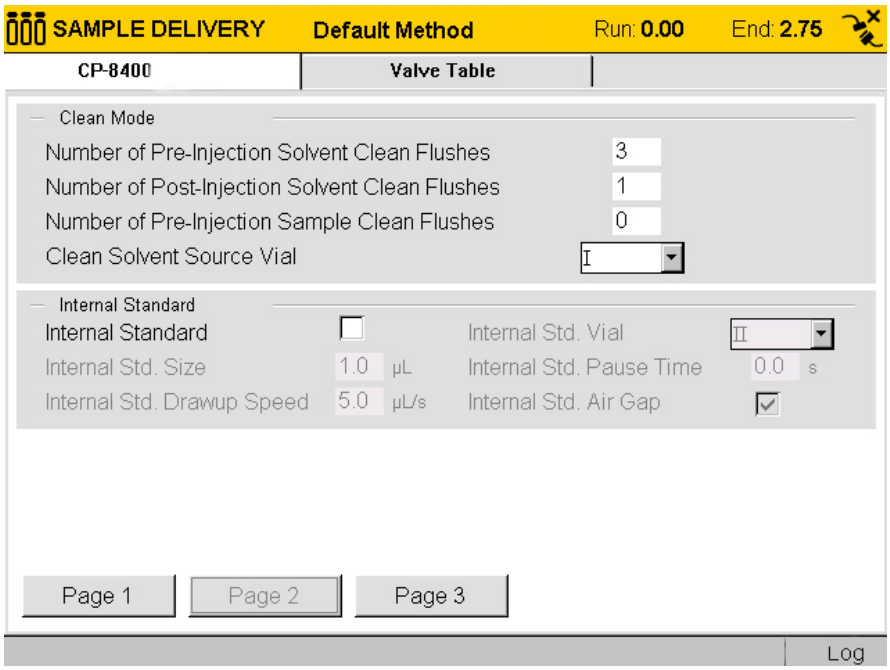
Fill Plunger Speed

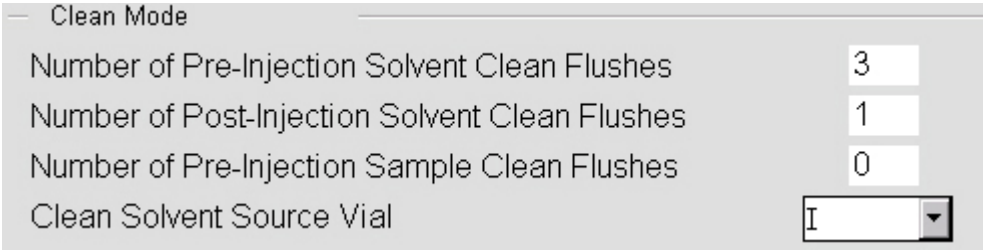
Defines the speed at which the plunger will be withdrawn to draw up sample. This parameter is required to prevent cavitation of some solvents during extraction.

Injection Plunger Speed

Depending on the volume of the syringe installed, this parameter will be limited to the specified ranges. The Injection rate setting has 0.1 µL/sec (depending of the syringe size) step resolution.

Page 2





Clean Mode

Number of Pre-Injection Solvent Clean Flushes (0-99)

This parameter defines the number of times the syringe will draw up solvent, rotate to the waste cup and expel the syringe contents before the sampling process begins. The default value is 3.

Number of Post-Injection Solvent Clean Flushes (0-99)

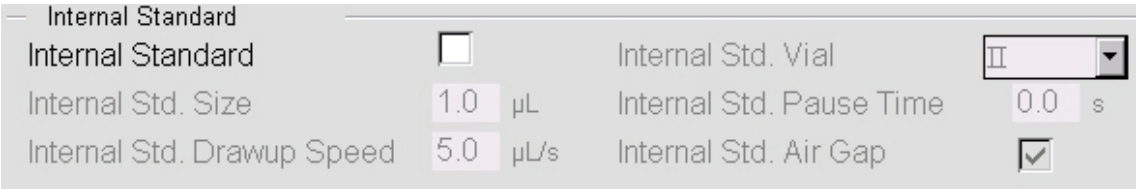
This parameter defines the number of times the syringe will draw up solvent, rotate to the waste cup and expel the syringe contents after the injection process has ended. The default value is 1.

Number of Pre-Injection Sample Clean Flushes (0-99)

This parameter defines the number of times the syringe will draw up sample, rotate to the waste cup and expel the syringe contents before the sampling process begins. The default value is 0.

Clean Solvent Source Vial (I, II, III, I&II, I&III, II&III, I&II&III)

There are three clean vials. This parameter selects which clean solvent source or combination of clean solvent sources the AutoSampler will use for its clean flushes. If a combination Clean Solvent Source is specified, the system will perform the number of solvent clean flushes as set by the operator from each of the solvent sources. Consequently, if the operator selected the Clean Solvent Source of I&II&III and the number of pre-injection solvent clean flushes was set to three, the system would draw up and expel solvent from Clean Solvent Source I three times, Clean Solvent Source II three times and finally Clean Solvent Source III three times for a total of nine syringe actions prior to beginning the sampling process. The default value for the Clean Solvent Source is I.



Internal Standard

Internal Standard

This parameter determines whether the system will use an Internal Standard as part of the sampling procedure. Selecting will cause the CP-8400/8410 to draw up the specified amount of Internal Standard from the specified vial.
The Internal Standard will always be withdrawn before the sample is withdrawn to avoid as much as possible cross contamination of the Internal Standard with analyses. The default value is blank.

Internal Std Size

This parameter defines the volume of Internal Standard that will be extracted for injection. The AutoSampler/AutoInjector will draw up the Internal Standard (depending of the syringe size) before the sample.

Internal Std Drawup Speed

This parameter defines how fast the plunger will be drawn out of the syringe extracting Internal Standard from the vial and into the syringe. The default value is 5.0 μL/sec.

Internal Std Vial (I, II, III)

This parameter will identify which of the three clean vials contains the Internal Standard. The AutoSampler/AutoInjector will draw up the Internal Standard before the sample. This order is to minimize contamination of the internal standard by residual sample. The default internal standard vial is Clean Vial position II.

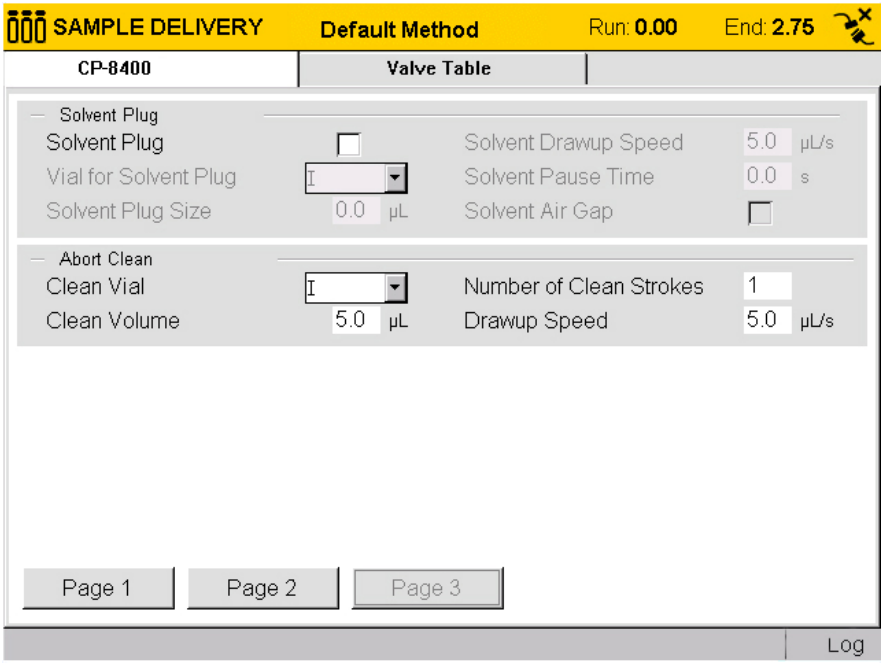
Internal Std Pause Time (0-9.9 sec)

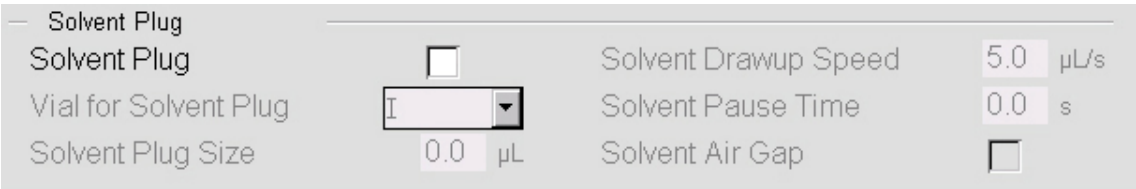
This parameter defines how long the syringe will remain in the vial after the plunger has reached its set withdrawal volume. This is required to allow the internal standard to fully migrate into the syringe. The default value is 0 sec.

Internal Std Air Gap

Selecting for this parameter will cause the AutoSampler to withdraw the plunger a distance equal to 1 μL (depending of the syringe size) after the syringe has been withdrawn from the vial. Selecting blank will cause the syringe plunger to remain in its position subsequent to the extraction of the Internal Standard until some other sampling action is required. No air gap will be withdrawn. The default value is checked.

Page 3





Solvent Plug

This parameter determines whether the system will us a solvent plug as part of the sampling procedure. Selecting will cause the CP-8400/8410 to draw up the specified amount of solvent from the specified vial. The default value is blank.

Vial for Solvent Plug (I, II, III)

This parameter defines which of the vials will be used as the source for the solvent plug. The default selection is wash vial I.

Solvent Plug Size

This parameter defines the volume of solvent that will be used for the solvent plug in 0.1 µL steps (depending of the syringe size) for the 5 and 10 µL syringes. The default volume is 0 µL.

Solvent Drawup Speed

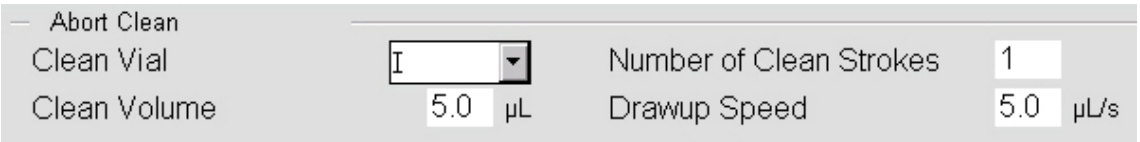
This parameter defines how quickly the plunger will be withdrawn form the syringe during the solvent plug extraction procedure. The default value is 5 µL/sec for each of the syringe types.

Solvent Pause Time (0-9.9 sec)

This parameter defines how long the syringe needle will remain in the vial after the plunger has been withdrawn a distance corresponding to its set volume in the Solvent plug size parameter. The default value is 0 sec.

Solvent Air Gap

Setting this parameter will cause the syringe plunger to be withdrawn an amount corresponding to 1 µL (depending of the syringe size) prior to the syringe sled descending and the solvent being drawn up. Setting this parameter to blank will cause no upper air gap to be formed. The default value is blank.



Abort Clean

Use in case Automation is stopped or a serious fault is detected.

Clean Vial (I, II, III)

This parameter specifies the wash solvent vial that will be used by the Default Cleaning procedure. The default value is 1.

Clean Volume

This parameter defines the amount of cleaning solvent that will be drawn up and expelled to waste. The volume is selectable in 0.1 µL (depending of the syringe size). The default value is 5.0 µL.

Number of Clean Strokes (0-10)

This defines the number of times the cleaning solvent will be drawn up and expelled into the waste cup. The default value is 1.

Clean Drawup Speed

This parameter specifies how quickly the plunger will be withdrawn from the syringe drawing solvent into the barrel. The default value is 5 μL/sec (depending of the syringe size). This same value should be used as the plunger speed when expelling the syringe contents into the waste cup.

SPME Sampling Mode

SPME mode is designed for using the special Solid Phase Micro-Extraction fibers and fiber holder.

WARNING

Do not use the clean mode actions unless you have experience with SPME or have very dirty samples with insoluble materials that need to be removed. Indiscriminate use of cleaning can introduce contaminants into your analytical run and degrade your results.

SPME mode actions are summarized below:

1. The number of pre-injection solvent clean flushes specified in the method will be performed by dipping the fiber into the solvent and waiting for each clean stroke the time specified in the Adsorb Time for Cleaning. Once the cleaning strokes are completed the tower will rotate over the waste station, the fiber holder will descend and the fiber exposed for drying/desorbing (desorbing requires a heated gas stream to be gently blown over the fiber).

2. If Prep-Ahead is used, the AutoSampler will begin the sampling process at the set delay time after the Prep-Ahead timer has expired. This allows you to sample for extended lengths of time without adversely affecting the throughput of your system. The Prep-Ahead timer starts after the Auto Sampler has finished all injections actions for the current injection.

3. If Derivatization is specified, the fiber will next be introduced into the Derivatizing Agent vial specified in the Derivatization page. The fiber will penetrate into the Derivatizing agent vial the depth set for the Solvent penetration depth on the main SPME parameters page. The fiber will wait for the specified Adsorb time and then be withdrawn.

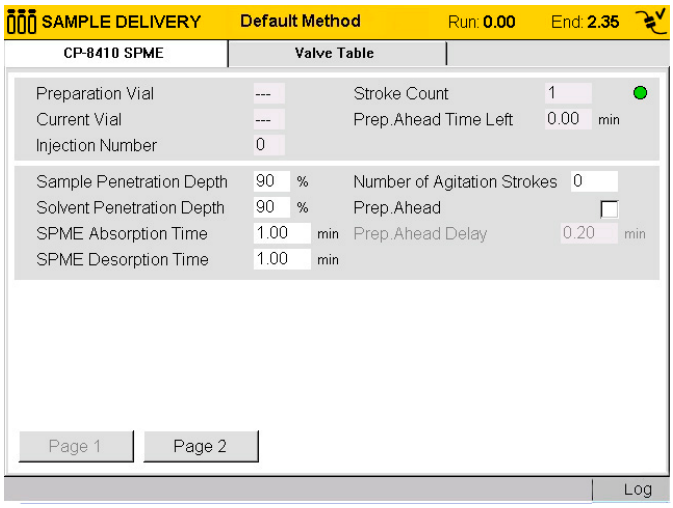
4. The SPME fiber holder will be positioned over the sample vial and the number of agitation strokes requested will be performed. Agitation is accomplished by moving the fiber into and out of the fiber holder at between 1 and 2 times per second. It is common for this action to pause a little longer sometimes relative to others. This non-regular motion is expected and normal. Each time the fiber descends into the sample, it will stay in the sample for the specified Adsorb Time. Use the Sample Penetration Depth to sample from either headspace (the vapor above the sample) or into the liquid. The fiber holder sheath (the needle at the end of the fiber holder) will penetrate about 6 mm into the sample vial. The fiber will be extended varying distances based on the penetration depth setting.

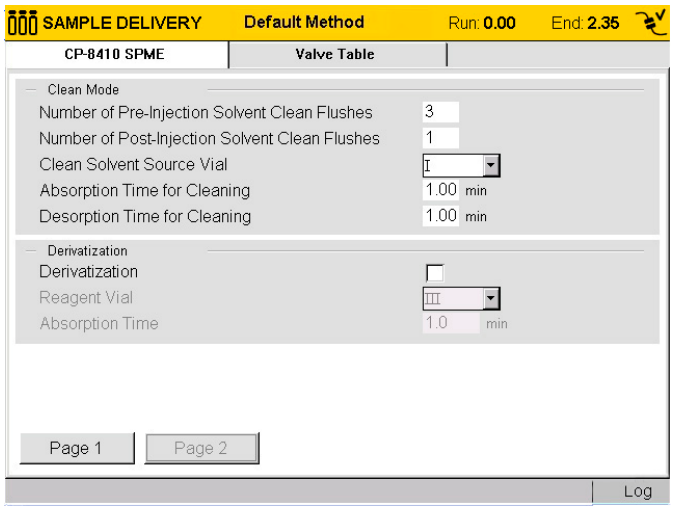
For liquid sampling you will get best results if the level of the liquid in the vial does not contact the fiber sheath thus potentially causing carry-over.

5. The AutoSampler will next perform the SPME sample introduction into the Injector. The SPME fiber holder will descend and penetrate the Injector septum. The fiber will then be extended the specified Desorb time on the main SPME parameters page.

6. Upon withdrawal from the Injector, any post injection clean actions will be performed.

The SPME mode screens and parameters are summarized below:





INSTALLATION OF THE SPME MODE

This section describes the installation of the SPME option on the CP-8400 AutoSampler and the CP-8410 AutoInjector.

Never use soaps, such as Snoop®. These may contaminate the system and cause permanent damage to capillary columns. If a liquid leak detector is desired, try a 50/50 mixture of isopropanol/water.

Familiarize yourself with the safety regulations and the general operating principles, including the calibration of the sampler positions.

To enable the sampler to perform SPME, 2 basic adjustments of the sampler are required:

- The syringe-holder bracket must be exchanged.
- The switch on the sampler tower, mounted on the sled, must be set in the SPME mode.

Figure 15: SPME Syringe and Holder

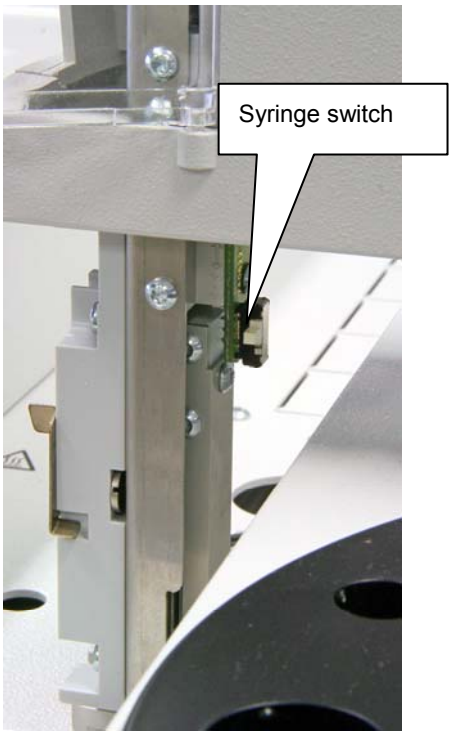
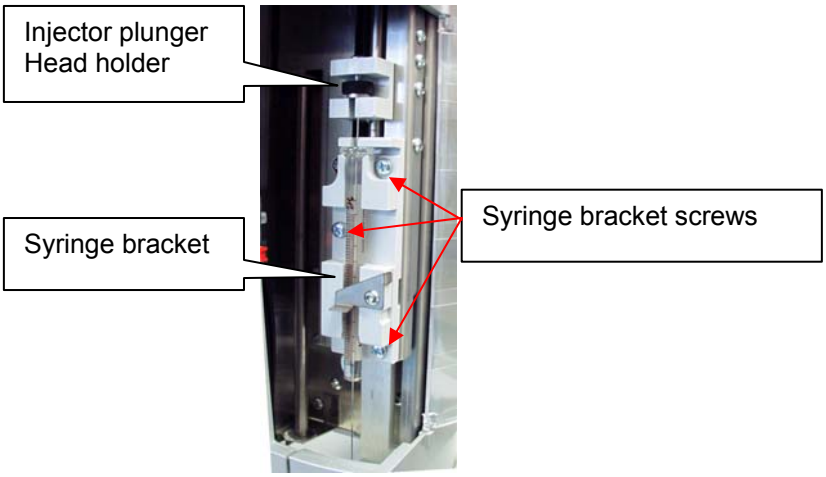


Figure 16: SPME/Standard Switch

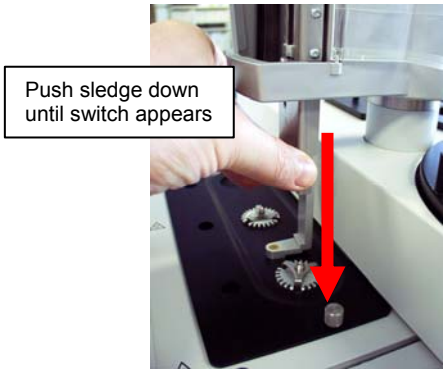
Installation of the SPME mode:

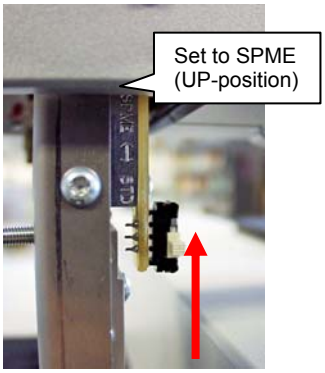
1. Turn off the main power switch and unplug the power plug.
2. Manually rotate the Sampler Tower so that it is over the GC. It is best not to place it over an Injector since the Injector may be hot.
3. Open de tower's clear plastic door and remove the syringe.
4. Remove the syringe bracket (3 screws).
5. Push the Injector's plunger-head holder downwards.



6. Push the syringe sled down as far as it will go.

Once the syringe-guide foot touches the top of the gas chromatograph, gently compress it further. Observe the white switch, which will be just exposed once the sled has reached its maximum downward travel.



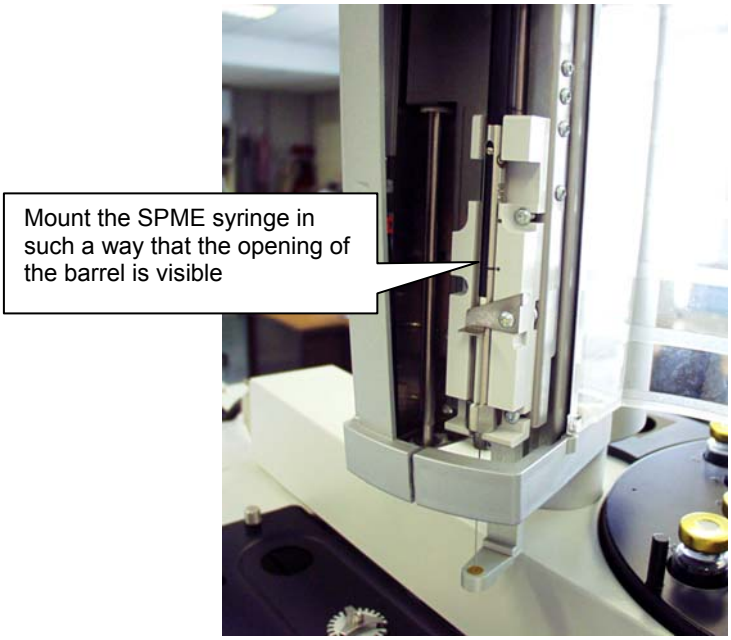


7. Set the switch to the SPME configuration (UP-Position).

8. Raise the sled until you hear a click, this fixes the sled.

9. Install the SPME syringe holder.

10. Install the SPME syringe, i.e. the fiber holder, in the block. Lock the plunger head and fix the syringe. Position the SPME syringe in such a way that you can see the opening in the barrel.



Installation of the fiber into the SPME syringe, i.e. the fiber holder, is explained in the fiber-holder manual.

11. Close the cover on the tower and turn on the power to the GC. The GC will automatically detect the new configuration of the switch and will respond accordingly.

12. Recalibrate the sampler, on page 112.

13. Adjust the Injector insert, on page 145 to SPME injection.

14. The instrument is now in the SPME mode.

Injector Insert

To assure optimum results when desorbing a SPME fiber, it is important to use the correct Injector insert. Use a straight, unpacked insert with an internal diameter of 0.8 mm (P/N: 392611948 for the 1079 Injector). Smaller inserts do not allow the fiber sheath to penetrate the Injector, while large inserts will broaden early-eluting analytes.

The SPME fiber is mounted in the fiber sheath. During septum penetration, the fiber is completely retracted into the sheath for protection. A blunt sheath with a relatively large diameter may result in septum leakage. If you encounter frequent septum failure, use the optional Merlin Microseal™.

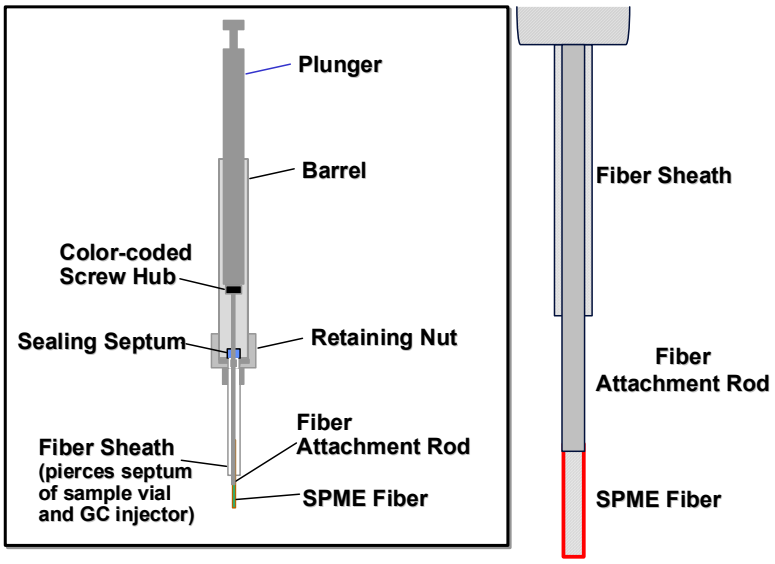


Figure 17: SPME Syringe

When performing liquid SPME, make sure that the fiber sheath does NOT contact the liquid. This may result in carry-over.

Injector Temperature

To accelerate the desorption process of the analytes from the SPME fiber, elevated injection temperatures are required. The ideal temperature is approximately 10-20°C below the maximum temperature of the fiber and GC column (usually between 200° and 280°C).

Sample Vials and Septa

SPME is a concentration technique normally used in combination with a GC/MS. To minimize pollution of the analysis by the vial septum, special Ultra clean septa are used. These vials are included in the SPME kit. Replacement parts see here, on page 441.

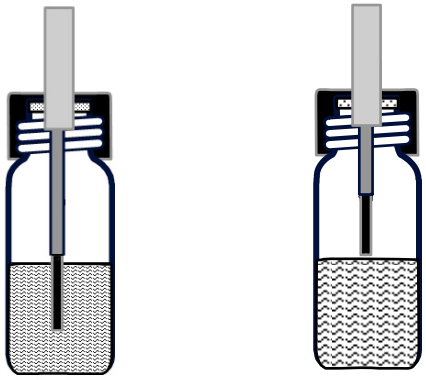
Agitation Strokes

SPME can be used in the agitation “mode”, which is useful when performing liquid SPME. Agitation disrupts the depletion zone that may surround the fiber during the extraction process. With CP-8400 and CP-8410 SPME, agitation is achieved by moving the fiber in and out of the sample liquid, approximately once every 2 seconds.

To use the agitation “mode”, fill in the number of Agitation Strokes (1 or more). Zero will disable the Agitation “mode”.

Sample Penetration Depth (%)

The penetration depth indicates the position of the fiber in the vial in the DOWN position. The UP position is a fixed value, which is 1 cm higher than the DOWN position. The DOWN position is set at the point at which the fiber is just fully immersed in the sample liquid.



Typical value for the 2 mL vial (P/N: 392611549 and P/N: 392611979)

- Sample volume 1 mL
- Penetration depth 60%

SPME Adsorption Time

The SPME adsorption time is the period between agitation strokes. If the number of agitation strokes is 0, the SPME adsorption time is equal to the sampling time.

Number of Agitation Strokes

The number of agitation strokes is the number of up and down movements of the fiber between the sample liquid and the space above the sample in the vial.

As a guideline, use the following formula to calculate the sampling time:

Sampling time (min) = $(t_{Adsorb} + t_{agitation}) * (n_{strokes} + 1)$

Whereby:
t_{Adsorb} is the adsorption time (min)
$t_{agitation}$ is the time per agitation stroke (approx. 0.01 min)
$n_{strokes}$ is the number of agitation strokes

For example: an adsorption time of 5 minutes, in combination with 10 agitation strokes will result in the following sampling time:

Sampling time = (5+0.01)*(10+1) is approx. 55 min

Optimum analytical yield results are obtained with an adsorption time that is set to 0. The sampling time is determined by the number of agitation strokes only.

INSTALLATION OF THE LIQUID SAMPLING MODE

This section describes the installation of the Liquid Sampling option on the CP-8400 AutoSampler and the CP-8410 AutoInjector.

Familiarize yourself with the safety regulations and the general operating principles, including the calibration of the sampler positions.

To enable the sampler to perform Liquid Sampling, 2 basic adjustments of the sampler are required:

- The syringe-holder bracket must be exchanged.
- The switch on the sampler tower, mounted on the sled, must be set in the STD (Liquid Sampling) mode.

Figure 18: Liquid Sampling Syringe and Holder

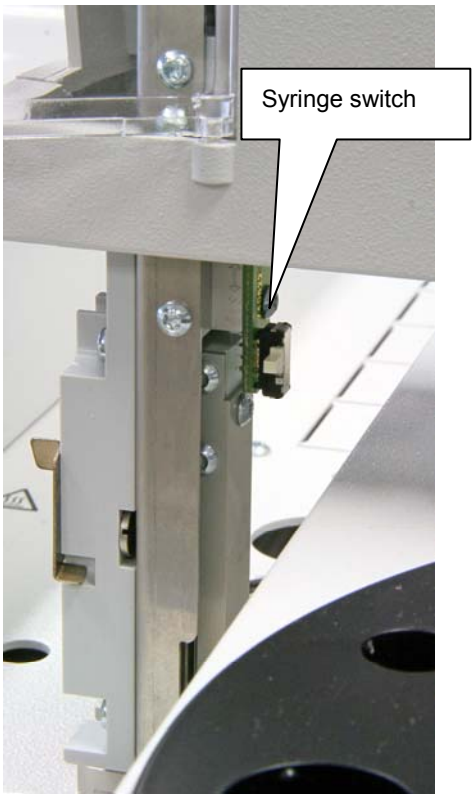
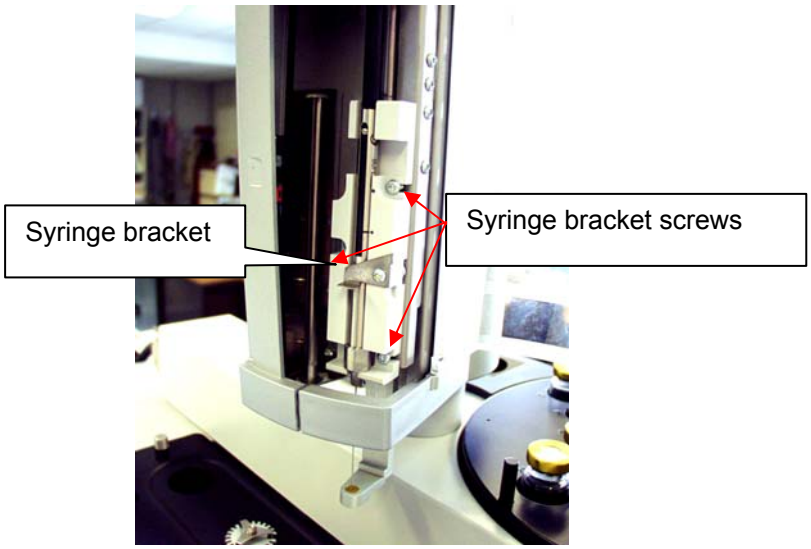


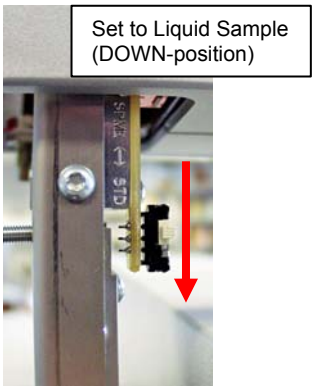
Figure 19: SPME/Standard Switch

Installation of the liquid sampling mode:

1. Turn off the main power switch and unplug the power plug.
2. Manually rotate the Sampler Tower so that it is over the GC. It is best not to place it over an Injector since the Injector may be hot.
3. Open de tower's clear plastic door and remove the syringe.
4. Remove the syringe bracket (3 screws).
5. Push the Injector's plunger-head holder downwards.



Once the syringe-guide foot touches the top of the gas chromatograph, gently compress it further. Observe the white switch, which will be just exposed once the sled has reached its maximum downward travel.

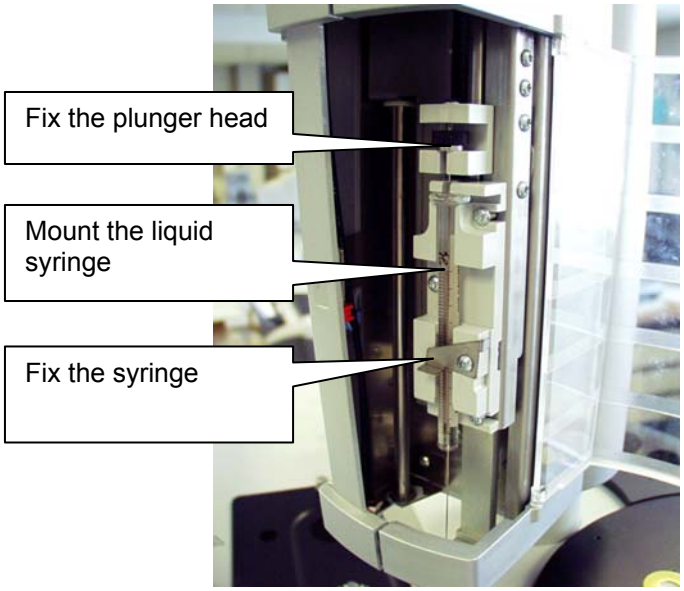


6. Set the switch to the STD (Standard, liquid sample configuration) DOWN-Position.

7. Raise the sled until you hear a click, this fixes the sled.

8. Install the Liquid Sample syringe holder.

9. Install the Liquid Syringe, in the block. Lock the plunger head and fix the syringe.



10. Close the cover on the tower and turn on the power to the gas chromatograph. The gas chromatograph will automatically detect the new configuration of the switch and will respond accordingly.

11. Recalibrate the sampler, on page 112.

12. Adjust the Injector insert to Liquid Injection.

13. The instrument is now in the Liquid Injection mode.

THE 1177 SPLIT/SPLITLESS CAPILLARY INJECTOR

The 1177 is a dedicated Split/Splitless Isothermal Capillary Injector which operates in 2 modes:

- Split.
- Splitless.

The 1177 Injector can be used with a wide range of narrow bore to wide bore (100 to 530 μm ID) capillary columns.

The basic pneumatics design is a wide range inlet flow controller and back pressure regulated column head pressure. This is the case whether manual pneumatics or Electronic Flow Control (EFC) is used.

Features of the 1177 Injector

- The 1177 operates isothermally from 50 to 450 °C.
- Changing from one injection mode to another typically involves a change of Injector insert and a modification to the Injector and EFC program. Glass inserts can be easily changed from the top of the Injector.
- The internal Injector temperature profile keeps the septum cool while maintaining the point of injection at the setpoint temperature.
- The 1177 uses positive septum purge to minimize the adsorption of sample onto the Injector septum and to prevent contaminants from the septum entering the column.
- The 1177 uses a unique dual split vent that allows effective sweeping of the entire Injector body.

Automatic Start Switch

The automatic start switch is a spring loaded actuator that fits over and is aligned with, the injection port of the 1177 Injector nut. The GC run is started when the actuator is depressed by the syringe barrel, or manually pressed at the moment of sample injection. The GC run can also be manually (only in local automation) started by pressing the START .

WARNING:
BURN HAZARD

The Injector nut and automatic start switch assembly may be very hot during instrument operation and should not be touched with unprotected hands.

1177 Injector Inserts

Click here to see the 1177 Injector Inserts, on page 448.
Note that all 1177 Injector inserts are deactivated for maximum inertness.

1177 Modes of Injection

The 1177 injection mode is defined by the Split Vent program and by the choice of insert. The Split Vent is controlled differently based on the pneumatics installed, if EFC the split valve is controlled from the Injector section, or in case of manual pneumatics the split valve (is controlled from the valve table). The following are brief descriptions of the various modes of injection. More detailed information on each mode is given later in this section.

Split Mode

The split injection mode is preferred for the analysis of relatively concentrated samples. The sample is split in the Injector with a representative portion entering the column. The split injection mode provides the shortest sampling time which leads to sharp chromatographic peaks. Use the 4.0 mm open insert, or the packed 4.0 mm ID insert when operating in the split mode.

In the split injection mode, the sample volume is typically 2 μL or less. Early eluting compounds usually appear as very sharp peaks. In some cases, the peak width is less than one second. It is important that you inject the sample as quickly as possible. If you are using the CP-8400 AutoSampler use the Standard Split/Splitless mode of injection. If you are using a non-Varian AutoSampler or you are using the User Defined Mode of operation on the CP-8400 AutoSamplers, if the sample injection time (the time between the insertion and removal of the syringe needle from the Injector) exceeds the peak width, peaks can broaden, tail or chromatographic performance will be degraded. With broader, later eluting peaks, it is less important that you inject the sample quickly. The split ratio (fraction of sample that enters the column) is the ratio of Splitvent flow to the column flow.

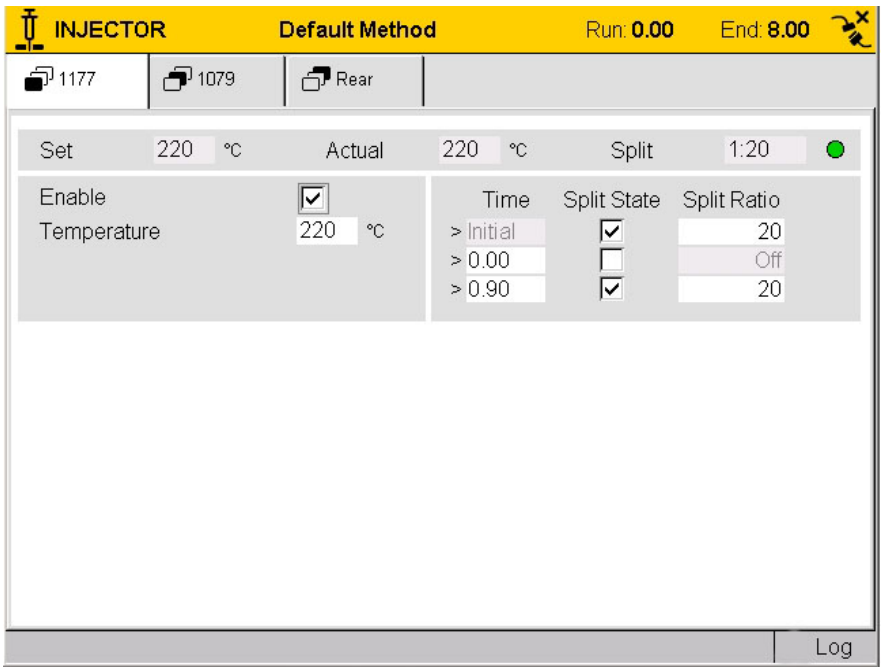
Splitless Mode

The 4 mm Goose-neck insert packed with glass wool is typically used for isothermal splitless injection. The small capillary section of the glass insert fits around the syringe needle and restricts backflush of the sample vapor during injection. In the splitless injection mode, the sample enters the column during a variable sampling time at the beginning of the analysis. This period is typically 30-90 seconds during which there is no flow from the Injector to the split vent. After the sampling time, the Injector is vented to remove any residual solvent and sample out of the Injector.

1177 Injector Screen

The 1177 Injector screen allows modifying the Injector parameters.
With the Enable checkbox it is possible to enable/disable the Injector temperature control.
In the Temperature box the wanted Injector temperature can be set.
The Split State controls the flow of carrier gas through the Injector during the analytical run. When the Split State is unchecked, most of the sample injected is directed onto the column. When the Split State is checked, the sample is split in the Injector with typically the smaller fraction entering the column and the larger fraction being vented.

In the example below is a splitless injection. The Injector is held in the split state for the initial period with split ratio of 20. When the run is started the split state will switched Off. After 0.9 minute the split is switched on again.



1177 Electronic Flow Control (EFC)

The EFC module used with an 1177 Injector is the EFC21 or EFC25.
The EFC21 and EFC25 type are designed specifically for the 1177 and 1079 PTV Injectors to support their various modes of operation. It duplicates the behavior of the Split/Splitless manual pneumatics system in that there is an inlet mass flow controller supplying carrier gas to the Injector and a pressure control valve downstream from the Injector which sets the Injector pressure. As Injector pressure determines the rate of carrier gas flow through the column, this pressure is monitored close to the point of injection.
Type EFC21 allows the user to set constant Injector pressure or constant flow. In addition the split ratio can be programmed.

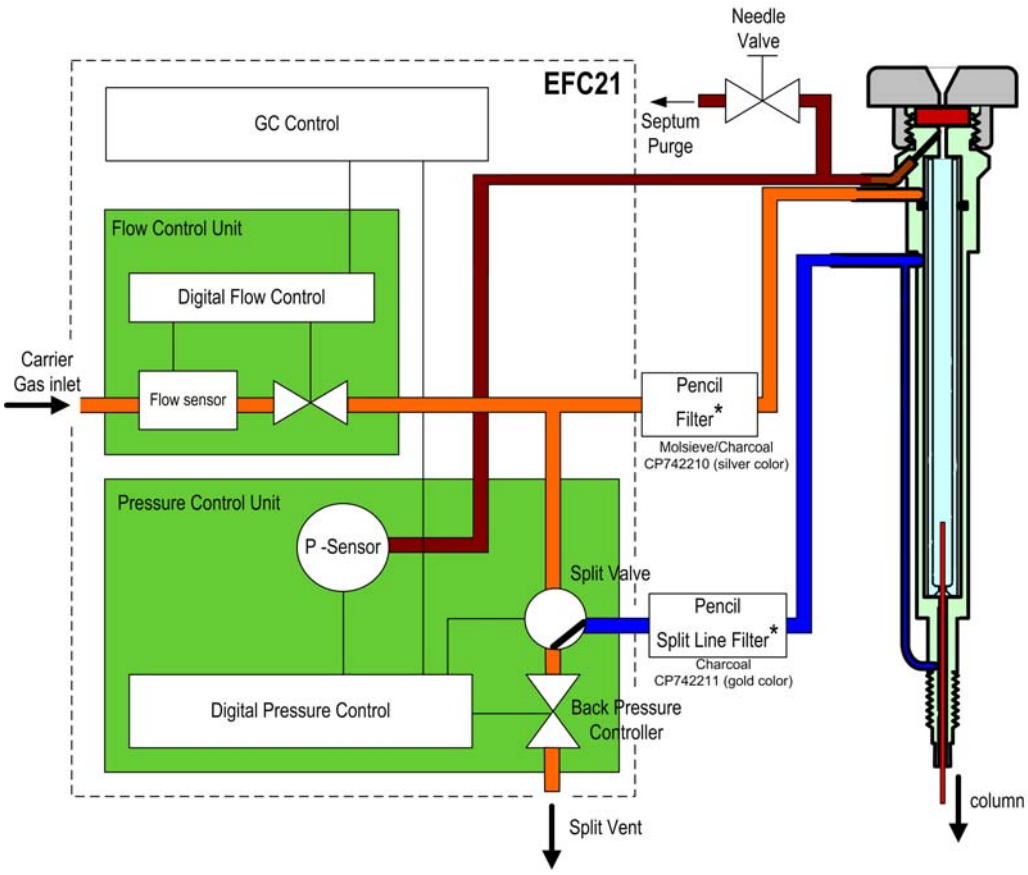


Figure 20: EFC21 and 1177 in Split mode Flow Diagram

* See Pencil Filters on page 437

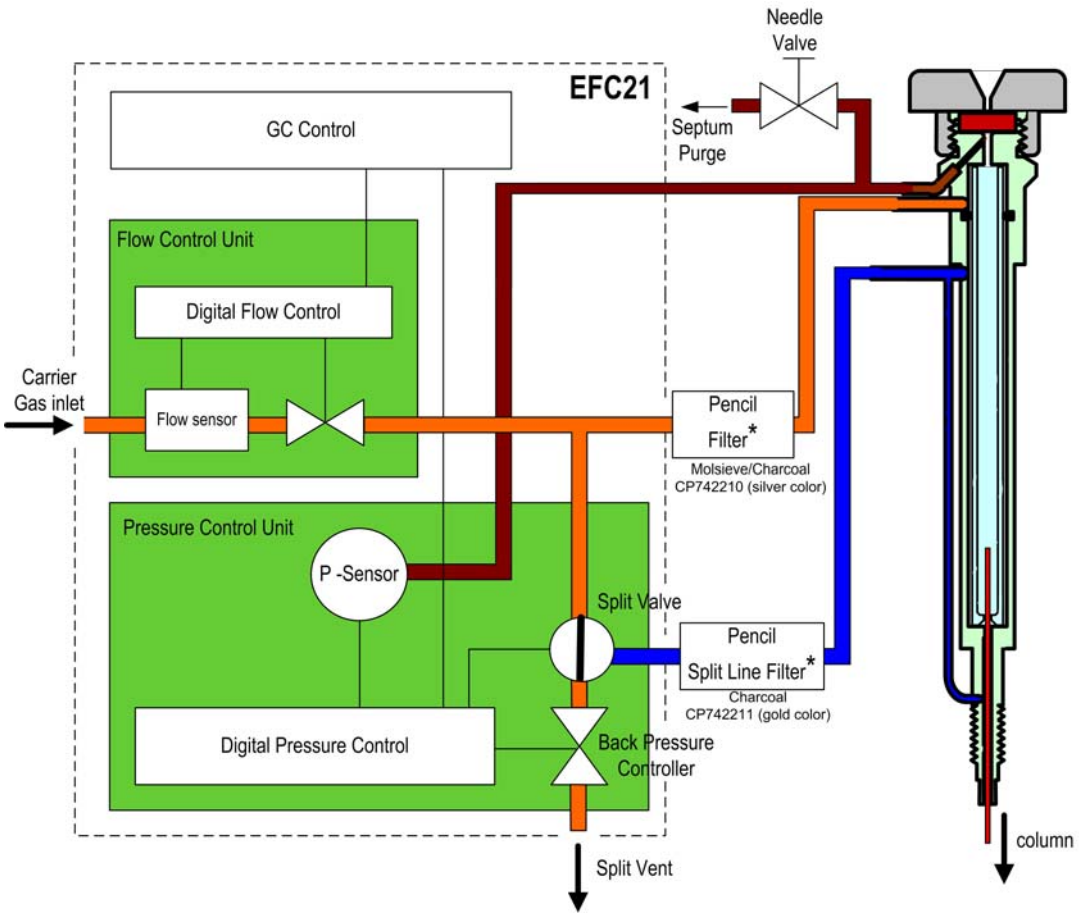


Figure 21: EFC21 and 1177 in Splitless mode Flow Diagram

* See Pencil Filters on page 437

The EFC25 is almost identical with the EFC21 the only exception is that the pressure is monitored at the module itself rather than at the Injector. This allows the EFC25 to be used with Purge and Trap devices between EFC and Injector.

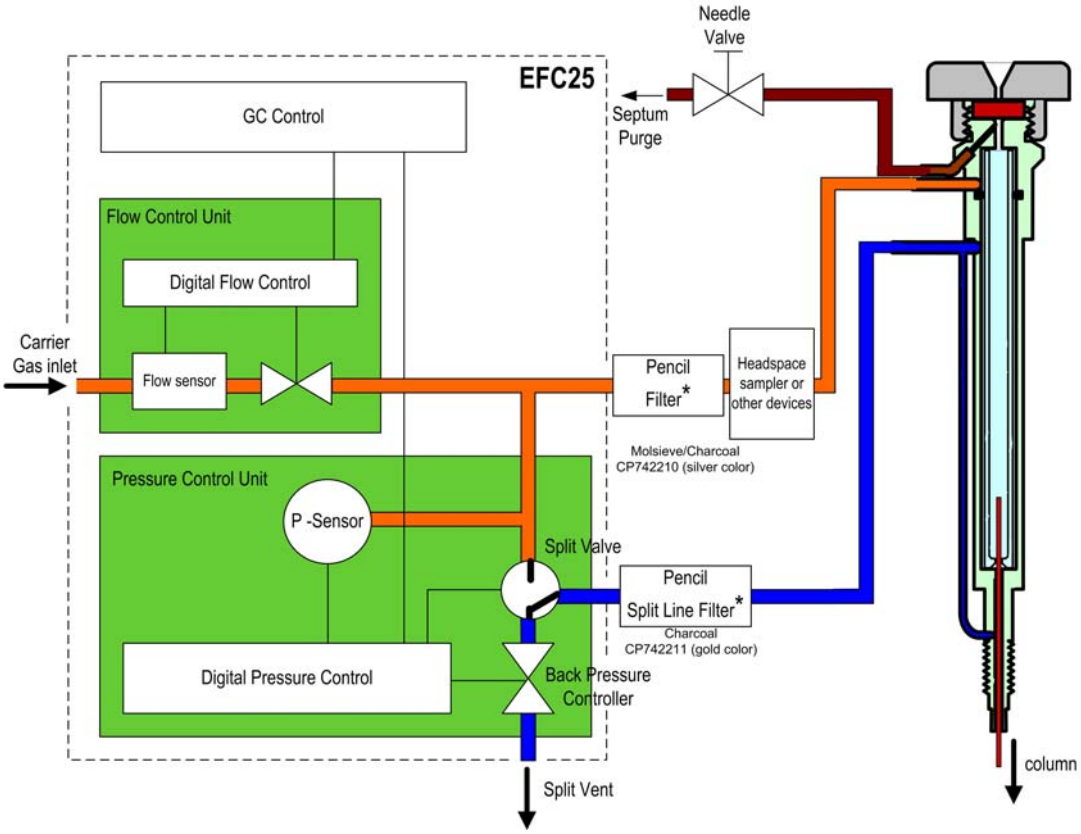


Figure 22: EFC25 and 1177 Flow Diagram

* See Pencil Filters on page 437

1177 Manual Pneumatics

The total flow into the system is adjusted using a 0–800 mL/min manual flow controller. The 1177 Injector uses a flow controller to supply the total flow into the system with a back pressure regulator to control the column head pressure. When the Injector is operated in the split mode the flow out of the split vent relative to the flow through the column is defined as the split ratio.

In the splitless mode, gas does not flow through the split line from the Injector to the 3-way solenoid valve (see Figure 21). Rather, gas from the flow controller by-passes the Injector to pass through the 3-way solenoid valve. The back pressure regulator uses this flow to control column head pressure, which in turn controls carrier gas flow through the column.

Operation of the 1177 Injector

The following section describes how to operate the 1177Split/Splitless Capillary Injector with the Varian 450-GC. It is separated into a series of procedures, starting with installation of a column and basic programming of the Injector from the keyboard, followed by detailed information on the various modes of injection.

Column Installation

The “Connect Capillary Column to Injector” on page 15 procedure describes the installation of a capillary column in a Varian 450-GC equipped with an 1177 Injector. Each step of the procedure is described in detail.

Condition the Column

For conditioning the capillary column see “Column Installation and Conditioning” on page 23.

Column Installation in Detector

For connecting a capillary column into a detector, see “Connect Capillary Column to Detector” on page 24.

Setting 1177 Gas Flow Rates

The gas flow rates for the 1177 Split/Splitless Capillary Injector can be set using manual pneumatics or Electronic Flow Control. Note that a positive flow through the column must be set before heating the column.

CAUTION

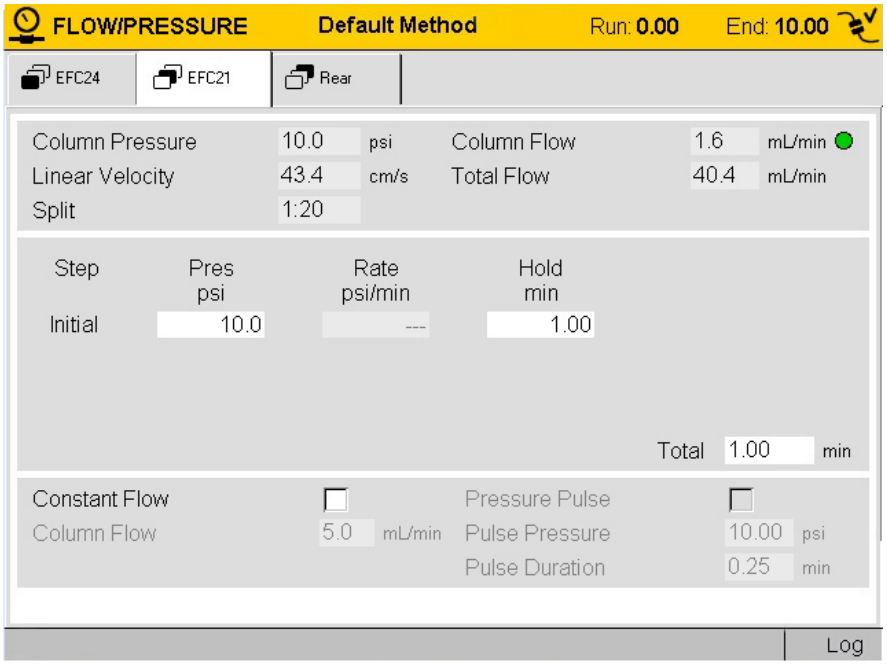
Do not heat the column oven above 50 °C without carrier gas flowing through the column. The column phase can be irreversibly damaged.

EFC Pneumatics

Type EFC21 or EFC25 used with the 1177 Injector allows the user to set a constant column head pressure, build a pressure program, or set a constant column flow. In addition, a split ratio can be set or time programmed. A pressure program is typically used to maintain the column flow at a constant value while temperature programming the column oven. When Constant Flow Programming is enabled, the pressure program needed to maintain constant flow is derived whenever the method is loaded.

The 1177 is a pressure-controlled Injector; thus the column flow decreases with increasing column temperature if the pressure remains constant. EFC method parameters and status are accessible via the FLOW/PRESSURE key on the 450-GC keyboard.

The Type EFC21 status field displays the actual column pressure (in the units chosen in Setup), calculated column flow rate, calculated column linear velocity and the split ratio. The split ratio status is either off if the split state is set to OFF, or a whole number.
The lower part allows you to view/edit constant flow mode.
The following screen is an example of a type EFC21 method.



Constant Column Flow

Type EFC21/EFC25 with the 1177 will automatically build a pressure program to keep the volumetric column flow rate constant during temperature programming of the column oven when Constant Flow is selected.

The same general guidelines should be followed for all injection modes. If the column is operated isothermally then the pressure should be kept constant. If the column is temperature programmed then the pressure can either be held constant or programmed. Programming the Injector pressure generally has no significant impact on chromatography, other than a slight reduction in analysis time. In some instances resolution may either improve or degrade depending on the settings. A technique called Pressure Pulse is also available for use with Constant Flow programs with Type EFC21/EFC25. Use the following guide for setting Injector pressure and/or building a pressure program.

Temperature programming the column oven results in an increase in carrier gas viscosity which results in a decrease in column flow rate. This effect can be offset by applying the appropriate column pressure program.

If Constant Flow Programming is enabled, a pressure program based on the column parameters and the column oven temperature program will be automatically rebuild parameter change result in an automatic rebuild of the pressure program.

When entering a pressure program to maintain constant column flow rate, the program is based on the column temperature program. If the column oven is operated isothermally, then constant pressure is maintained to achieve a desired flow rate.

Pressure Pulse

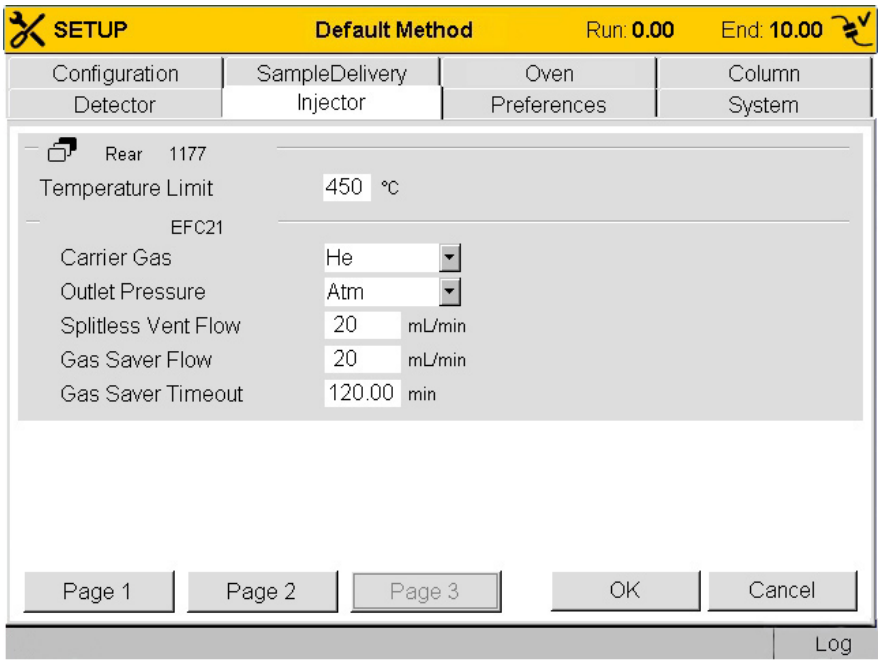
Once you have selected Constant Flow programming, a checkbox “Pressure Pulse” becomes accessible.

Selecting Pressure Pulse gives you the option of enabling a pressure pulse, setting the desired pressure and the duration for which the pressure should last. Typically, the pressure pulse pressure is held for between 30 and 90 seconds. The purpose of the pressure pulse is twofold. The higher pressure will prevent the solvent vapor cloud from becoming excessively large allowing larger injection volumes and providing more efficient passage of the sample into the column. The higher pressure also causes higher flow rates into the column making the transfer quicker and thus preventing excessive residence times in the Injector with consequent decomposition of labile compounds. **If you use Pressure Pulse you need to perform a septum purge calibration using the higher pressure pulse pressure.**

- The GC will go ready based on the pressure as defined in pressure pulse.
- When a run is started the column pressure as defined in pressure pulse will remain for the “Pulse Duration” time. After the pulse duration time the column pressure is calculated from the constant flow conditions.

Gas Saver

For type EFC21 and EFC25 a specific Gas Saver Flow and a timeout period may be set. Press the setup key and click on the Injector tab now choose the correct Injector (Page 1 or Page 2 or Page 3).



A time entry of 0 turns the Gas Saver feature off. If a non-zero time is entered, the Gas Saver timer will start incrementing. If a run is not started or a new method is not loaded or download by Workstation, the set Gas Saver flow rate will be activated (the instrument will go into stand-by). A message will be logged in the instrument log. **Stand-by is canceled by loading a method or pressing the end stand-by key.**

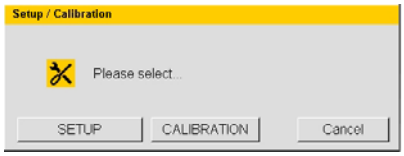
Septum Purge Calibration

The 450-GC is equipped with a septum purge for the Model 1177 split/splitless Injector and is controlled by a manual needle valve. This is located behind the front cover of the GC.

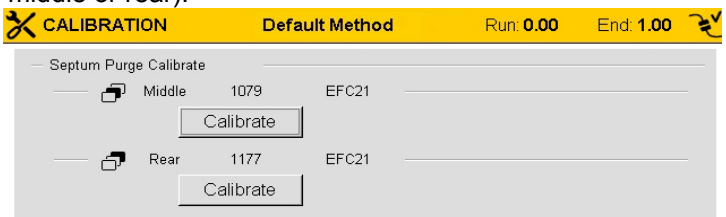
The manual needle valve can offer advantages over fixed restriction types as it can be adjusted to suit the more demanding applications. For the accurate display of total column flow and velocity it does require calibration when columns of different length and diameter are installed.

With type EFC21, the septum purge calibration routine should be carried out when the instrument is first set up or when a new column is installed or when a significantly different pressure point is chosen.

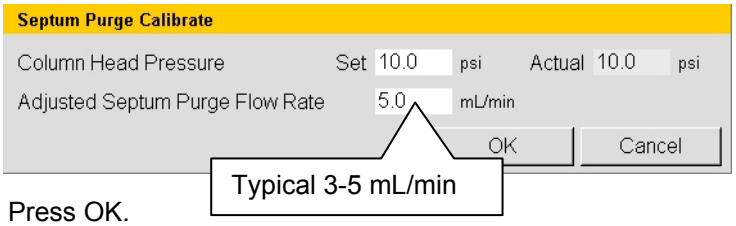
Press the SETUP key and click on Calibration.



The Calibration page will appear, choose Calibrate for the correct Injector (front, middle or rear).



Enter the desired Column Head pressure. After a few seconds measure the septum purge flow rate and adjust the septum purge valve (located behind the column oven door) to the desired flow, enter this value (typical 3-5 mL/min) in the Adjusted Septum Purge Flow Rate field.



Press OK.

Manual Pneumatics

Tools and equipment needed

- Bubble or electronic flowmeter.

1. Turn the 1177 Split Flow Controller (on the GC pneumatics panel) counterclockwise to open the split flow controller.

2. Adjust the Back Pressure Regulator (on the GC pneumatics panel) to establish a positive column head pressure (monitored on the pressure gauge).

Set the column head pressure based on the column installed in the GC. For example, for a 30m x 250 µm ID column, set the column head pressure to 12-15 Psig to achieve ~1 mL/min column flow rate at 50 °C oven temperature (helium).

3. Connect the flowmeter to the split vent on the left side of the GC and measure the split vent flow rate. Turn the Split Flow Controller valve to adjust the split vent flow rate to 50 mL/min.

4. Adjust the Septum Purge Needle valve to adjust the septum purge flow rate to 3 - 5 mL/min. Readjust the split vent flow rate to 50 mL/min.

5. Before heating the column, purge the system with carrier gas for 10 - 15 minutes.

With manual pneumatics the split valve should be configured in the sample delivery/valve table in set-up of the 450-GC. Split/splitless mode can be programmed through the valve table in the sample delivery page of the GC method.

1177 Modes of Operation

The 1177 can be operated in several modes, depending on the nature of the sample and requirements of the analysis. When designing an injection method the most important parameters are:

- The Injector insert used.
- The Injector and column temperature when the injection is made.
- The carrier gas flow profile through the Injector.

The following is a brief description of the method parameters used for each injection mode. In all cases Electronic Flow Control (EFC) is used for carrier gas control.

In many cases switching from one 1177 mode to another involves changing the glass insert. A detailed stepwise procedure to carry out this task is given in the Maintenance section of this manual, on page 171. In most injection modes the insert is installed with an O-ring.

Split Injection

The split mode is used when samples are relatively concentrated and for neat samples. This mode of injection involves rapid vaporization of the sample followed by sample splitting. Splitting involves directing a portion of the sample into the column while the remainder is vented. The split ratio is defined as the proportion of sample vented to the sample entering the column. With EFC carrier gas control this parameter can be set automatically in the 1177 method.

Injector Insert	4 mm ID open insert packed with glass wool.
Column Installation	3.7 cm from the bottom of the column nut at the base of the Injector.
Injector Temperature	Isothermal 250 °C.
Column Temperature	50 °C initial for 0.1 min, ramp to 250 °C at 20 °C/min, hold 5 min.
Carrier Gas Control	Set the split mode to ON for the duration of the run in the 1177 method section and set the split ratio to 100. The sample is split upon injection and a representative portion representing 1/100th of the amount injected enters the column. Note that the split ratio is a method specific parameter and should be set appropriately for individual analyses. In addition the Injector pressure or pressure ramp should be set to achieve the desired column flow rate.

Table 29: Split Injection Typical Conditions

Splitless Injection

The classical splitless injection technique involves vaporizing the sample in a hot Injector and slow transfer to the column. The split state is OFF during the sampling period; therefore, all of the injected sample should enter the column. The initial column temperature is maintained for at least the sampling time to trap all sample components at the head of the column. At the end of the sampling period (typically 0.5 to 1.5 minutes) the split state is turned ON to vent any residual sample or solvent from the Injector.

The following table describes typical method parameters for an isothermal splitless injection.

Injector Insert	4 mm ID open insert packed with glass wool.
Column Position	3.7 cm from the bottom of the column nut at the base of the Injector.
Injector Temperature	Initial temperature isothermal 250 °C.
Column Temperature	50 °C initial for 1 min, ramp to 250 °C at 20 °C/min, hold 5 minutes.
Carrier gas control	Set the initial split mode to OFF and time program it to ON after 0.75 minutes. In this case the splitless sampling time is 0.75 minutes. The split ratio during the split ON period should be set to 50. Set the appropriate pressure or pressure ramp to achieve the desired column flow rate.

Table 30: Splitless Injection Method Parameters

Note that in all the above cases the parameters given are generic and need optimization (including column position) for specific applications. Particular care should be taken with the large volume mode of injection where the initial Injector temperature and timing of the split states have to be carefully selected.

Testing the 1177 Injector Performance

The following procedure describes how to test the performance of the 1177 Capillary Injector. This is best done with a test sample containing an appropriate set of components for the installed detector. The following table lists the series of test samples available for 450-GC.

Test Sample	*Part Number*	*Concentrations of Test Compounds*
TCD	82-005048-01	3.00 μg/μL of C_{14}, C_{15} and C_{16} in iso-octane.
ECD	82-005048-02	33.0 pg/μL of lindane and aldrin in iso-octane.
PFPD	82-005048-03	20.0 ng/μL of n-dodecanethiol, tributylphosphate, methyl parathion; 4000 ng/μL of n-pentadecane in iso-octane.
TSD	82-005048-04	2.00 ng/μL of azobenzene, methyl parathion; 4.00 ng/μL malathion and 4.00 μg/μL C_{17} in iso-octane.
FID	82-005048-07	30 ng/μL of C_{14}, C_{15} and C_{16} in iso-octane.
Note: *If the FID test sample is not available, the TCD test sample can be used if first diluted 100:1.*		

Table 31: Detector Test Samples

To run one of these test samples, use the chromatographic conditions listed previously for the injection technique you are currently using. The detector should be operated at the most sensitive range, e.g., 12 for FID and TSD, 0.05 for TCD, 10 for PFPD and 1 for ECD. The resultant chromatogram on page 419 should approximate that shown in the detector section of this manual.

1177 Injector Assembly

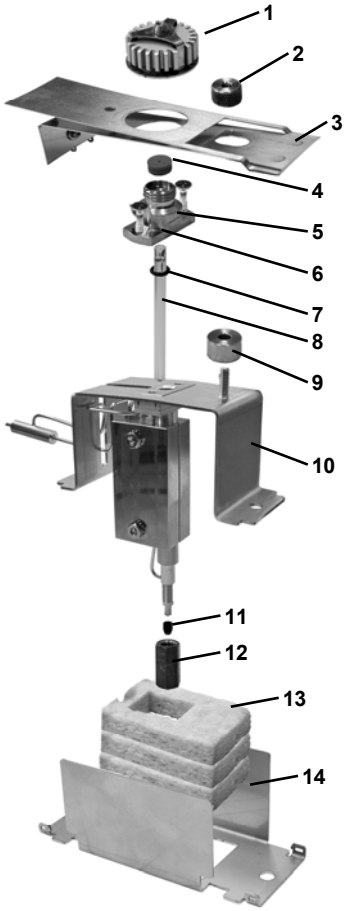


Figure 23: 1177 Injector Assembly

1. Injector Nut (392597501)
   Injector Nut Wrench (390842300)
2. Knob (392597101)
3. Inject Switch Assembly (390820601)
4. Septum, 9 mm
   BTO (lowest bleed, CR298713)
   Marathon (Autosampler, CR239778)
   Advanced Green 3 (general purpose, CR246713)
   Septum pick (7200008400)
5. Septum Purge Head

Pneumatic Type	*Stainless Steel*	*UltiMetal*
EFC21	392597301	392597303
EFC25 or Manual Pneumatics	392597302	392597304

6. Purge head screw (2x 391866308)
7. O-ring, Liner
   Graphite, 6.5mm for Splitless (392611930)
   Viton, 6.3mm (8850103100)
8. Spacer (39258101)
9. Glass Insert (default 392611927[1] More liners)
10. Injector Body
    Stainless Steel (392599401)
    UltiMetal (392599411)
    Manual (392599501)
11. Column Ferrule (see table below)
12. Capillary Column Nut (394955100)
13. Insulation
14. Cover

Column ID	*Holes*	*Teflon* *Max 250°C*	*Vespel* *Max 350°C*	*40% Graphite 60% Vespel* *Max 400°C*	*Graphite* *Max 450°C*	*SilTite* *Metal, GC/MS*
0.18 mm ID and smaller	1	CR214103	CR212103	CR213103	-	-
0.25 mm ID	1	CR214104	CR212104	CR213104	CR211104	-
	2	-	CR212124	CR213124	CR211124	-
0.25 mm ID and smaller	1	-	-	-	-	SG073300
0.32 mm ID	1	CR214105	CR212105	CR213105	CR211105	SG073301
	2	-	CR212125	CR213125	CR211125	-
0.53 mm ID	1	CR214108	CR212108	CR213108	CR211108	SG073302

Table 32: 1177 Column Ferrules 1/16"

[1] In combination with the PFPD detector the Siltek liner (RT210462145) must be used.

Maintenance

Before maintaining the 1177 Injector please read the common Injector/septum maintenance information in the section Maintenance, on page 432.

The 1177 is a Split/Splitless capillary Injector. Typically, to change from Split operation to Splitless involves changing the Injector insert. Also replace the insert on routine base. This is especially important when dirty samples are analyzed.

Tools Required

- Tweezers or septum pick (P/N 72-000084-00).
- Injector nut wrench (P/N 03-908423-00).
- Philips screwdriver (long handle).
- Clean laboratory tissue.

WARNING: BURN HAZARD

The Injector nut may be hot. Lower the Injector temperature to 50°C and permit the Injector nut to cool before proceeding.

Remove the Glass Insert

Follow these steps to remove the glass insert from the 1177 Injector.

1. Use the Injector nut wrench to remove the Injector nut. Place the nut on a clean surface (e.g., clean tissue).

2. Remove the inject switch by unscrewing the retaining nut.

3. Unscrew the 2 captive screws holding the top of the Injector to the base. Carefully move this assembly upwards and to one side. The Septum purge line and carrier gas supply lines may hinder movement of the top piece somewhat.

When using a CP-8400 AutoSampler, remove the Injector access plate by removing the 2 retaining screws and firmly lifting the plate. It may be hot if another Injector is also installed and powered. The plate may be a little tight, do not force it off. Lift straight up, a slight back and forth rocking may help remove it.

4. Use a laboratory tissue to grasp the glass insert and remove it from the Injector body.

Replace the Glass Insert

Follow these steps to replace the glass insert in the 1177 Injector.

1. Using tweezers or clean lab tissue, carefully slide the new insert into the Injector body. Place a new seal over the insert.

2. Carefully place the top of the Injector over the insert. Tighten the 2 captive screws alternately for uniform sealing. Note that placing the top piece onto the body may take some care since the gas supply line and septum purge lines may have minor resistance to movement.

3. Install a new septa, place the Injector nut on the Injector and tighten by hand until you feel some resistance, then tighten an extra 1/4 turn using the Injector nut wrench.

After the Injector nut has been replaced, check the split vent and septum purge flow rates to ensure these values have not changed.

4. Condition the insert by setting the 1177 Injector to the split mode and purging with carrier gas for 30 min at 300°C.

Injector body cleaning

To clean the Injector body, proceed as follows:

1. Set the column oven temperature to 45 °C and the 1177 Injector temperature to 50 °C. Wait for the zones to reach their set temperatures and then turn the column oven off by unchecking the checkbox “Power” located in the Oven screen .

2. Use the 5/16” open-end wrench to loosen the capillary column nut.

3. By hand, carefully withdraw the fused silica column and nut from the Injector assembly. Set the column nut and column end on the floor of the GC oven.

WARNING:
CHEMICAL HAZARD

WARNING:
FIRE HAZARD

Use proper eye and skin protection. Methanol and acetone are toxic and flammable chemicals. Exercise appropriate precautions when these chemicals are used.

4. Remove the Injector nut, septum and top of Injector.

5. Remove the Insert.

6. Moisten a cotton swab and a stick with methanol and gently swab center of the Injector body.

7. Moisten a cotton swab on a stick with iso-octane and gently swab the center of the Injector body.

8. Replace the insert, seal, Injector top, septum and Injector nut.

9. Examine the end of the fused silica column to ensure that it has not been damaged. Slide the end of the fused silica column into the opening of the Injector.

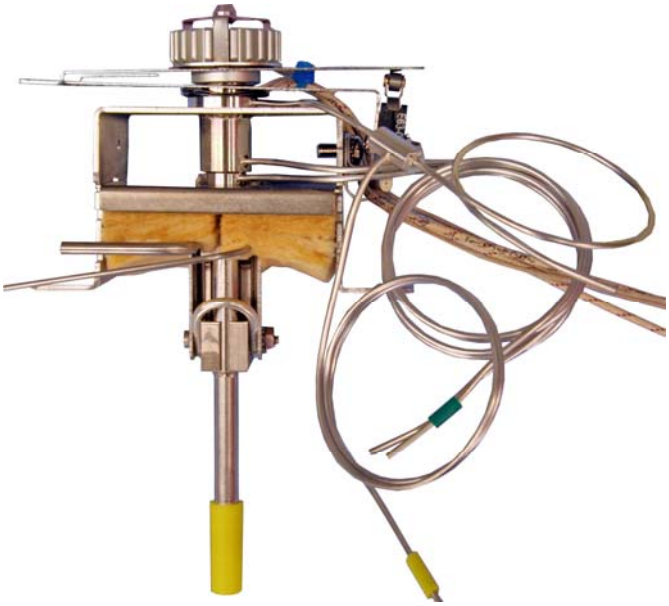
10. Gently twist the column nut in the opposite direction used to tighten the nut. Place the column nut on the end of the Injector and tighten the nut. This procedure minimizes twisting the column.

11. Use the 5/16″ open-end wrench and tighten with 1/6-turn to secure the column in place.

12. To check whether the Injector is leak-tight, connect a flow meter to the split vent. Set the Relay to the split mode. If the flow meter indicates a flow less than previously measured, the Injector has a leak.

Turn the column oven back on by checking the checkbox “Power” in the oven screen .

THE 1079 PTV INJECTOR



The 1079 is optimized as Programmable Temperature Vaporizing (PTV) Injector and is also used for large volume injections (LVI).
The 1079 PTV represents the latest evolution in Varian's Injector technology - extending the benefits of Varian's patented 1093 Septum-equipped Programmable Injector (SPI), to include split and splitless injection.
The 1079 PTV delivers improved chromatographic performance and increased GC up-time, is compatible with the full range of open tubular columns and is capable of isothermal or temperature programmed operation.

Advanced Performance
Five modes of operation: split, splitless, temperature-ramped splitless, Large Volume Injection (LVI) and cold on-column; allow maximum Injector flexibility.

- Temperature-programmable on-column mode optimizes the recovery of polar and thermolabile compounds.
- Large-volume injections in temperature-ramped splitless mode means less sample preparation and increased sensitivity.
- Manual or electronic control of gas flow guarantees optimum retention time reproducibility.
- An insert sealing design enables easy insert removal and replacement.
- A unique inlet design for unparalleled system inertness allows analysis of even the most thermolabile compounds.

Features of the 1079 PTV Injector

- The 1079 PTV can be operated isothermally or temperature programmed. The temperature range of the Injector is from -99 to 450 °C. Sub-ambient temperatures are achieved using cryogenic cooling.

- Changing from one injection mode to another typically involves a change of Injector insert and a modification to the Injector program. Glass inserts can be easily changed from the top of the Injector.

- Temperature programming in the splitless mode gives better recovery of labile analytes and is useful for wide boiling point mixtures.

- The Injector temperature profile keeps the septum cool while maintaining the point of injection closer to the setpoint temperature.

- The 1079 PTV Injector design facilitates large volume injection (5 - 250 µL).

- The 1079 PTV uses positive septum purge to minimize the adsorption of sample onto the Injector septum and to prevent contaminants from the septum entering the column.

- The 1079 PTV combined with the ChromatoProbe on page 198 is an effective tool for chromatographic analysis of dirty samples (liquids, solids and slurries, blood, urine or milk), without cleanup or extraction. The ChromatoProbe is based on intra-Injector thermal extraction of the semi volatile compounds in a sample microvial, while non-volatile residue is retained in the microvial, which is disposed of after the analysis.

Automatic Start Switch

The automatic start switch is a spring loaded actuator that fits over and is aligned with, the injection port of the 1079 PTV Injector nut. The GC run is started when the actuator is depressed by the syringe barrel, or manually pressed at the moment of sample injection. The GC run can also be manually (only in local automation) started by pressing the START .

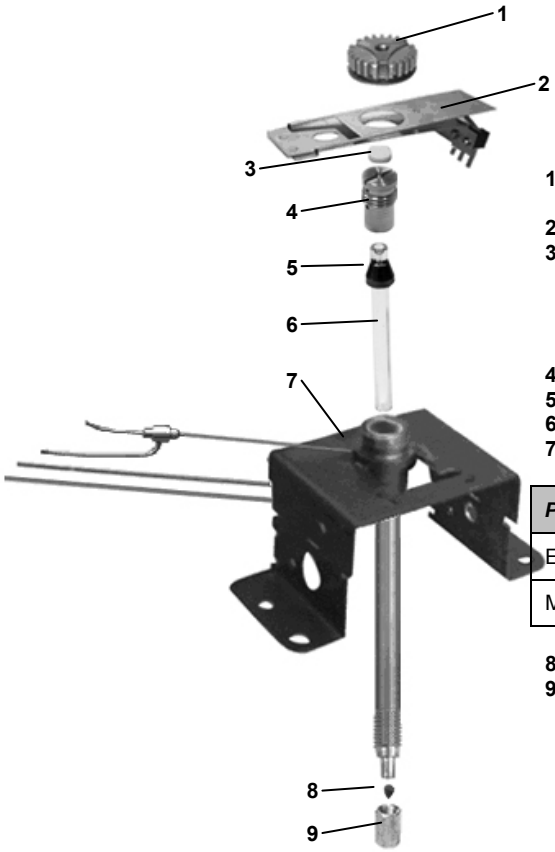
WARNING: BURN HAZARD

The Injector nut and automatic start switch assembly may be very hot during instrument operation and should NOT be touched with unprotected hands.

1079 PTV Injector Inserts

Click here to see the 1079 PTV Injector Inserts, on page 451.
Note that all 1079 PTV Injector inserts are deactivated for maximum inertness.

1079 PTV Injector Assembly



1. Injector Nut (394966601)
   Injector Nut Wrench, (390842300)
2. Automatic Start Switch (390820601)
3. Septum, 11.5 mm
   BTO (lowest bleed, CR298717)
   Marathon (Autosampler, CR239787)
   Advanced Green 3 (general purpose, CR246725)
   Septum pick, 7200008400
4. Septum Support (391867600)
5. Insert Ferrule (392534201)
6. Glass Insert, default 392611945 More liners
7. Injector Body

Pneumatic Type	*Stainless Steel*	*UltiMetal*
EFC	392544001	392544011
Manual Pneumatics	392559601	392559611

8. Column Ferrule, see table below
9. Bottom Nut (394955100)

Figure 24: 1079 PTV Injector Assembly

Column ID	*Holes*	*Teflon* *Max 250°C*	*Vespel* *Max 350°C*	*40% Graphite 60% Vespel* *Max 400°C*	*Graphite* *Max 450°C*	*SilTite* *Metal, GC/MS*
0.18 mm ID and smaller	1	CR214103	CR212103	CR213103	-	-
0.25 mm ID	1	CR214104	CR212104	CR213104	CR211104	-
	2	-	CR212124	CR213124	CR211124	-
0.25 mm ID and smaller	1	-	-	-	-	SG073300
0.32 mm ID	1	CR214105	CR212105	CR213105	CR211105	SG073301
	2	-	CR212125	CR213125	CR211125	-
0.53 mm ID	1	CR214108	CR212108	CR213108	CR211108	SG073302

Table 33: 1079 Column Ferrules 1/16"

1079 PTV Modes of Injection

The 1079 PTV injection mode is defined by the Split Vent program and by choice of insert. The Split Vent is controller differently based on the pneumatics installed, with EFC the split valve is controlled from the Injector section, with manual pneumatics the external events control the split valve. The following are brief descriptions of the various modes of injection; more detailed information on each mode is given later in this section. The 1079 PTV is optimized for PTV and LVI, but it is possible that he Injector can work in other modes.

Temperature Ramp Splitless Mode

The splitless temperature ramp mode is preferred for compounds that are altered by higher temperatures (thermolabile). Also, the splitless temperature ramp mode is used with wide boiling range mixtures (e.g., hydrocarbon mixtures). 2 glass inserts are recommended when operating in the splitless temperature ramp mode: 1) The 2 mm ID glass wool packed insert is used for non-polar compounds at levels >1 ng; and 2) the 0.5 mm ID open insert is used for thermolabile and/or polar compounds at trace levels (pg level).

In the splitless temperature ramp mode, the Injector is held at a temperature that is equal to or slightly below the boiling point of the injection solvent. The sample is deposited on the surface of the insert. After injection, the temperature of the Injector is increased rapidly (ramped). As the temperature of the Injector increases, the sample then vaporizes and is swept onto the column.

Large Volume

In the large volume mode of injection typically > 5 µL of sample is deposited into the Injector slowly, the solvent is vented and then the components of interest are transferred to the column. This is done using a special split vent program and an Injector temperature ramp. The CP-8400/8410 AutoSampler allows automated injection of up to 250 µL of sample.

Split Mode

The split injection mode is preferred for the analysis of relatively concentrated samples. The sample is split in the Injector with a representative portion entering the column. The split injection mode provides the shortest sampling time which leads to sharp chromatographic peaks. Use the 3.4 mm open insert, the 3.4 mm ID fritted insert, or the packed 3.4 mm ID insert when operating in the split mode.

In the split injection mode, the sample volume is typically 1 µL or less. Early eluting compounds usually appear as very sharp peaks. In some cases, the peak width is less than one second. Thus, it is important that you inject the sample as quickly as possible. If the sample injection time, (time between the insertion and removal of the syringe needle from the Injector) exceeds the peak width, peaks can broaden, tail or chromatographic performance will be degraded. With broader, later eluting peaks, it is less important that you inject the sample quickly. The split ratio (fraction of sample that enters the column) is the ratio of the flow of carrier gas out the split vent to the flow through the column. The 1177 Split/Splitless Injector on page 153 is optimized for Split application.

Isothermal Splitless Mode

The 3.4 mm insert packed with glass wool is typically used for isothermal splitless injection. The small capillary section of the glass insert fits snugly around the syringe needle and restricts backflush of the sample vapor during injection. In the splitless injection mode, the sample enters the column during a variable sampling time at the beginning of the analysis. This period is typically 30 - 90 seconds during which there is no flow from the Injector to the split vent. After the sampling time, the Injector is vented to clean any residual sample out of the Injector. The 1177 Split/Splitless Injector on page 153 is optimized for Splitless application.

On-Column Modes

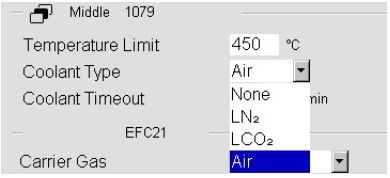
In the on-column modes the column is sealed to the glass insert (0.18 - 0.53 mm columns). This ensures that there is maximum transfer of sample to the column. In the on-column modes the sample is normally injected at or below the solvent boiling point and then the Injector is ramped to transfer the sample onto the column. The 1093 OC Injector, on page 231 is optimized for On-Column applications. Especially for applications operating at an oven temperature range that require short wire-bore columns.

1079 Injector Screen

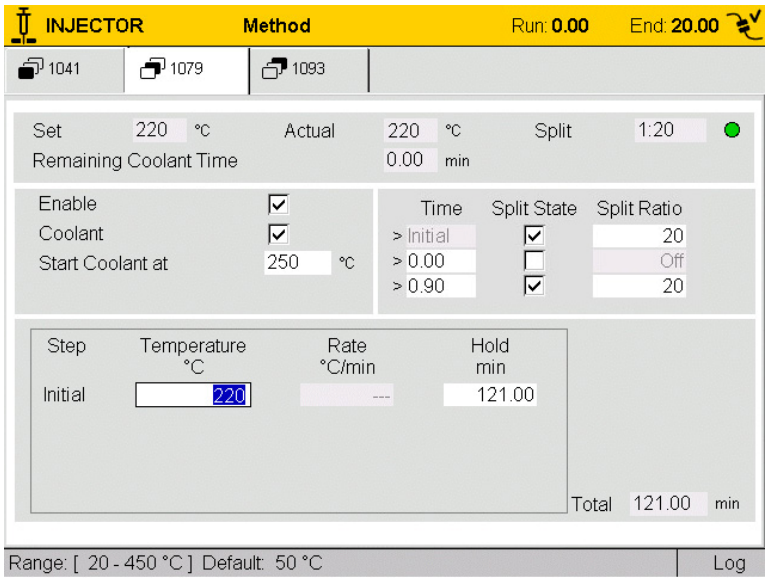
The 1079 Injector screen allows modifying the Injector parameters.
With the Enable checkbox it is possible to enable/disable the Injector temperature control.
In the Temperature box the wanted Injector temperature can be set.
The Split State controls the flow of carrier gas through the Injector during the analytical run. When the Split State is unchecked, most of the sample injected is directed onto the column. When the Split State is checked, the sample is split in the Injector with typically the smaller fraction entering the column and the larger fraction being vented.

In the example below is a splitless injection. The Injector is held in the split state for the initial period with split ratio of 20. When the run is started the split state will switched Off. After 0.9 minute the split is switched on again.

The Coolant checkbox turns on or off the cryogenic supply to the Injector, if installed. In the example below the cooling will start when the Injector temperature reaches the 250 °C. In the setup screen of the Injector tab 1079 some rarely changed coolant parameters can be changed, like coolant type (LN_2, LCO_2 or Air) and coolant timeout.



Coolant time-out refers to the period of time for which the coolant will remain on after it is turned on. For example, if the set temperature has not been reached or the run has not started within the specified time, the coolant supply will be turned off.



1079 PTV Electronic Flow Control (EFC)

The Electronic Flow Control module used on a 1079 PTV Injector is identified as a type EFC21 or EFC25. In simple terms it duplicates the behavior of the 1079 PTV manual pneumatics system in that there is an inlet mass flow controller supplying carrier gas to the Injector and a pressure control valve downstream from the Injector which sets the Injector pressure. As Injector pressure determines the rate of carrier gas flow through the column, this pressure is monitored close to the point of injection. The type EFC21 flow diagram shown below is an indication of the control mechanism of this type of EFC module.

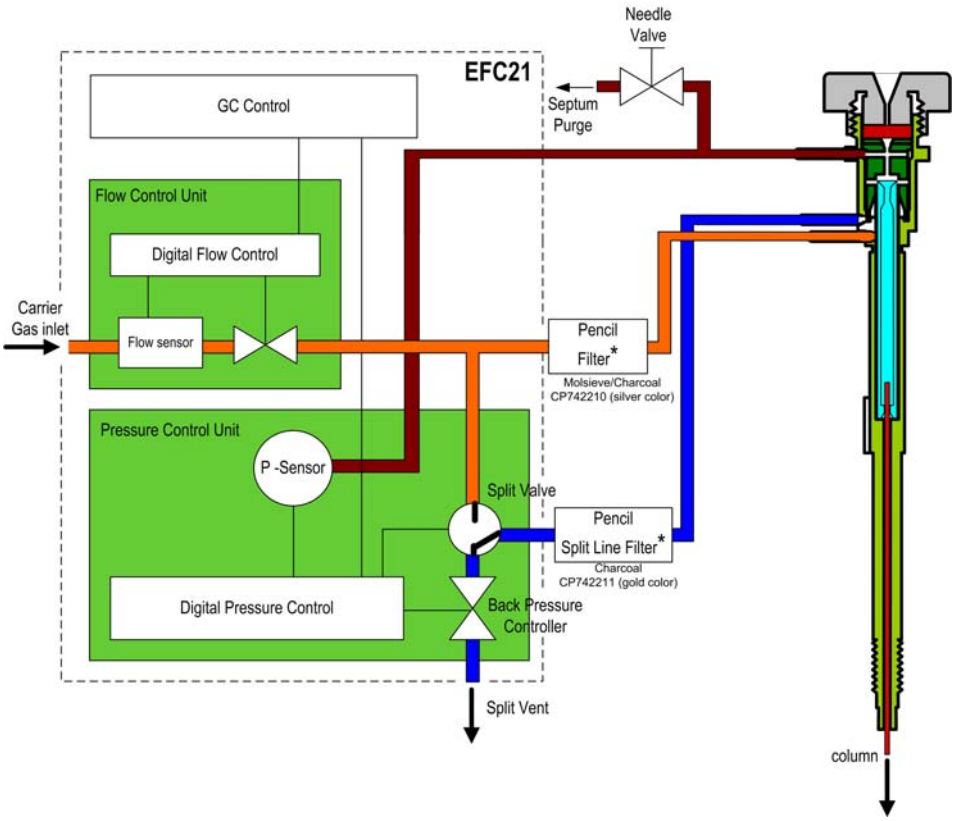


Figure 25: EFC21 and 1079 in Split mode Flow Diagram

*Pencil Filters on page 437

Type EFC21 allows the user set constant Injector pressure or constant flow. In addition the split ratio can be programmed.

The EFC25 is almost identical as the EFC21 the only exception is that the pressure is monitored at the module itself rather than at the Injector. This allows the EFC25 to be used with Purge and Trap devices upstream from the Injector.

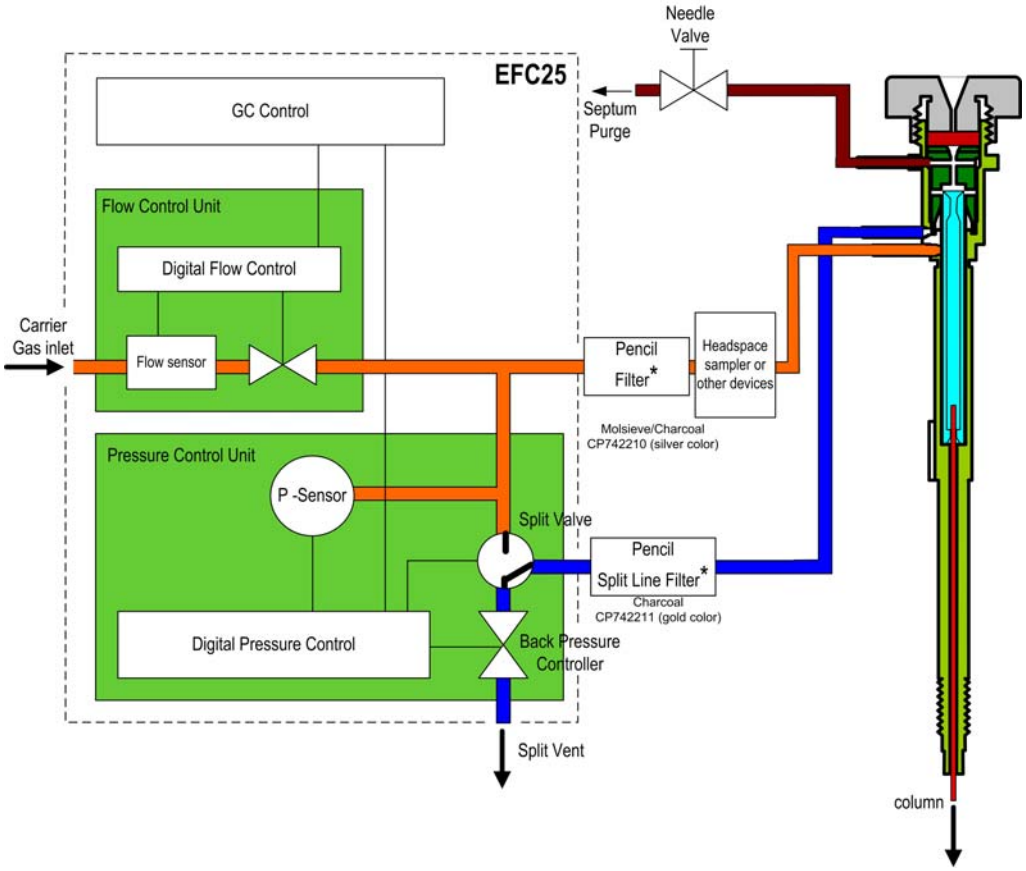


Figure 26: EFC25 and 1079 Flow Diagram

* See Pencil Filters on page 437

1079 PTV Manual Pneumatics

The total flow into the system is adjusted using a 0–800 mL/min manual flow controller. The 1079 PTV Injector uses a flow controller to supply the total flow into the system with a back pressure regulator to control the column head pressure. When the Injector is operated in the split mode the flow out the spilt vent relative to the flow through the column is defined as the split ratio.

In the splitless mode, gas does not flow through the split line from the Injector to the 3-way solenoid valve. Rather, gas from the flow controller by-passes the Injector to pass through the 3-way solenoid valve. The back pressure regulator uses this flow to control column head pressure, which in turn controls carrier gas flow through the column.

Operation of the 1079 PTV Injector

The following section describes how to operate the 1079 PTV Injector with the Varian 450-GC. It is separated into a series of procedures, starting with installation of a column and basic programming of the Injector from the keyboard, followed by detailed information on the various modes of injection.

Column Installation

The Connect Capillary Column to Injector on page 15 procedure describes the installation of a capillary column in a Varian 450-GC equipped with a 1079 PTV Injector. Each step of the procedure is described in detail.

Condition the Column

For conditioning the capillary column see Column Installation and Conditioning on page 23.

Column Installation in Detector

For connecting a capillary column into a detector, see Connect Capillary Column to Detector on page 24.

Setting 1079 PTV Gas Flow Rates

The gas flow rates for the 1079 PTV Injector can be set using manual pneumatics or Electronic Flow Control. Note that a positive flow through the column must be set before heating the column.

CAUTION

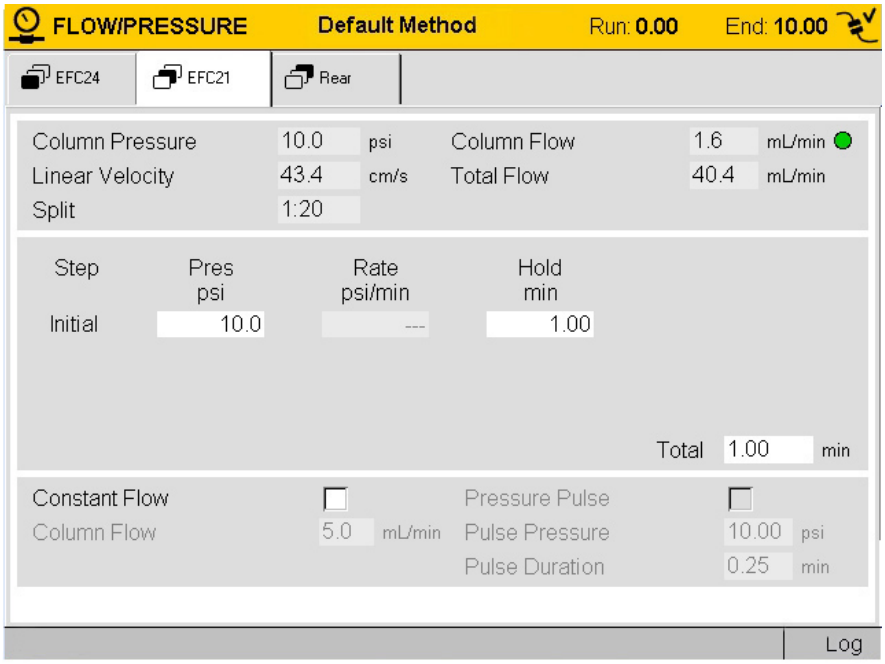
Do not heat the column oven above 50 °C without carrier gas flowing through the column. The column phase can be irreversibly damaged by exposure to oxygen at elevated temperatures. Allow 10 – 15 minutes to purge the column before heating.

EFC Pneumatics

Type EFC21 used with the 1079 PTV Injector allows the user to set a constant column head pressure, build a pressure program, or set a constant column flow. In addition, a split ratio can be set or time programmed. A pressure program is typically used to maintain the column flow at a constant value while temperature programming the column oven. When Constant Flow Programming is enabled, the pressure program needed to maintain constant flow is derived whenever the method is loaded.

The 1079 PTV is a pressure-controlled Injector; thus the column flow decreases with increasing column temperature if the pressure remains constant. EFC method parameters and status are accessed via the FLOW/PRESSURE key on the 450-GC keyboard.

The Type EFC21 status field displays the actual column head pressure (in the units chosen in Setup), calculated column flow rate, calculated column linear velocity and the split ratio. The split ratio status is either off if the split state is set to OFF, or a whole number.
The lower part allows you to view/edit constant flow mode.
The following screen is an example of a type EFC21 method.



Constant Column Flow

Type EFC21 with the 1079 PTV builds automatically a pressure program to keep the column flow rate constant during temperature programming of the column oven when Constant Flow is selected.

The same general guidelines should be followed for all injection modes. If the column is operated isothermally then the pressure should be kept constant. If the column is temperature programmed then the pressure can either be held constant or programmed. Programming the Injector pressure generally has no significant impact on chromatography, other than a slight reduction in analysis time.

In some instances resolution may either improve or degrade depending on the settings. For larger volume hot splitless injections, a technique called Pressure Pulse is also available for use with Constant Flow programs with Type EFC21. Use the following guide for setting Injector pressure and/or building a pressure program.

Temperature programming the column oven results in an increase in carrier gas viscosity which results in a decrease in column flow rate. This effect can be offset by applying the appropriate column pressure program.

If Constant Flow Programming is enabled, a pressure program based on the column parameters and the column oven temperature program will be automatically built. Any parameter change result in an automatic recalculation of the pressure program.

When entering a pressure program to maintain constant column flow rate, the program is based on the column temperature program. If the column oven is operated isothermally, then constant pressure is maintained to achieve a desired flow rate.

Pressure Pulse

Once you have selected Constant Flow programming, a checkbox “Pressure Pulse” becomes accessible.

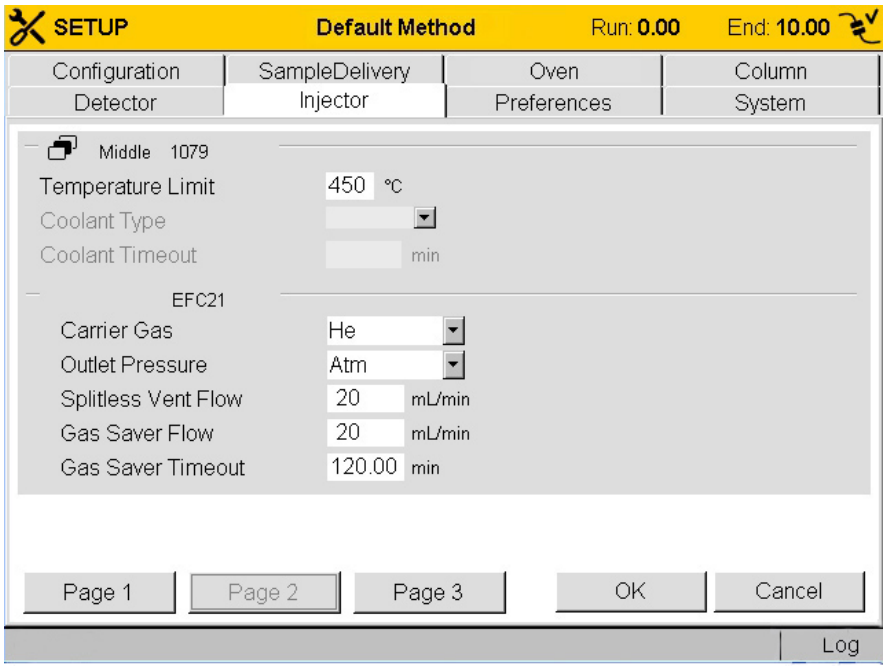
Selecting Pressure Pulse gives you the option of enabling a pressure pulse, setting the desired pressure and the duration for which the pressure should last. Typically, the pressure pulse pressure is held for between 30 and 90 seconds. The purpose of the pressure pulse is two fold. The higher pressure will prevent the solvent vapor cloud from becoming excessively large allowing larger injection volumes and providing more efficient passage of the sample into the column. The higher pressure also causes higher flow rates into the column making the transfer quicker and thus preventing excessive residence times in the Injector with consequent decomposition of labile compounds. **If you use Pressure Pulse you will need to perform septum purge calibration using the higher pressure pulse pressure.**

- The GC will go ready based on the pressure as defined in pressure pulse.
- When a run is started the column pressure as defined in pressure pulse will remain for the “Pulse Duration” time. After the pulse duration time the column pressure is calculated from the constant flow conditions.

Gas Saver

For type EFC21 a specific Gas Saver Flow and a timeout period may be set.

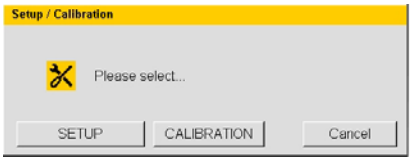
Press the setup key and click on the Injector tab, choose the correct Injector (Page 1 or Page 2 or Page 3).



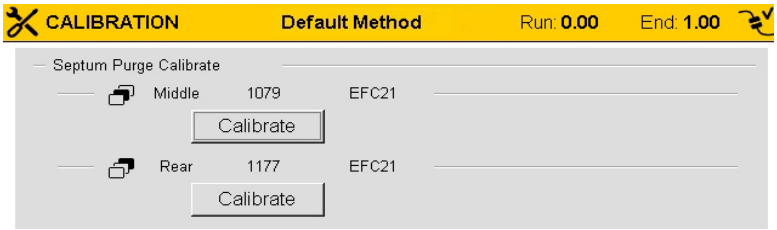
A time entry of 0 turns the Gas Saver feature off. If a non-zero time is entered, the Gas Saver timer will start incrementing. If a run is not started or a new method is not loaded or download by Workstation, the set Gas Saver flow rate will be activated (the instrument will go into stand-by). A message will be logged in the instrument log. **Stand-by is canceled by loading a method or pressing the end stand-by key.**

Septum Purge Calibration

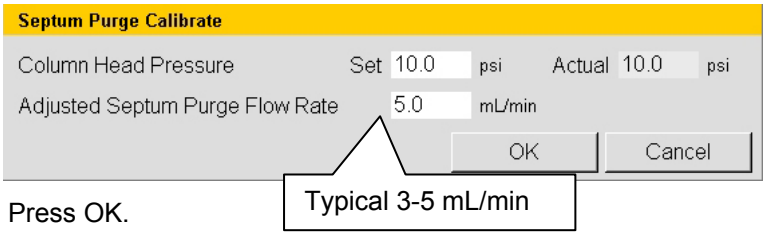
The 450-GC is equipped with a septum purge for the Model 1079 PTV Injector and is controlled by a manual needle valve. This is located behind the front cover of the GC. The manual needle valve can offer advantages over fixed restriction types as it can be adjusted to suit the more demanding applications. For the accurate display of total column flow and velocity it does require calibration when columns of different length and diameter are installed.
With type EFC21, the septum purge calibration routine should be carried out when the instrument is first set up or a new column is installed or when a significantly different pressure point is chosen. Press the SETUP key and click on Calibration.



The Calibration page will appear, choose Calibrate for the correct Injector (front, middle or rear).



Enter the desired Column Head pressure. After a few seconds measure the septum purge flow rate and adjust the septum purge valve (located behind the column oven door) to the desired flow, enter this value (typical 3-5 mL/min) in the Adjusted Septum Purge Flow Rate field.



Press OK.

Manual Pneumatics

Tools and equipment needed

- Bubble or electronic flowmeter, e.g., Varian Intelligent Digital Flowmeter, 01-900115-00.

1. Turn the 1079 PTV Split Flow Controller (on the GC pneumatics panel) counterclockwise to open the split flow controller.

2. Adjust the Back Pressure Regulator (on the GC pneumatics panel) to establish a positive column head pressure (monitored on the pressure gauge).

Set the column head pressure based on the column installed in the GC. For example, for a 30m x 250 µm ID column, set the column head pressure to 12-15 psig to achieve ~1 mL/min column flow rate at 50 °C oven temperature (helium).

3. Connect the flowmeter to the split vent on the left side of the GC and measure the split vent flow rate. Turn the Split Flow Controller valve to adjust the split vent flow rate to 50 mL/min.

4. Adjust the Septum Purge Needle valve to adjust the septum purge flow rate to 3-5 mL/min. Readjust the split vent flow rate to 50 mL/min.

5. Before heating the column, purge the system with carrier gas for 10 - 15 minutes.

1079 PTV Modes of Operation

The 1079 PTV can be operated in several modes, depending on the nature of the sample and requirements of the analysis. When designing an injection method the most important parameters are:

- The Injector insert used and the position of the column within the insert.
- The Injector and column temperature when the injection is made.
- The carrier gas flow profile through the Injector.

The following is a brief description of the method parameters used for each injection mode. In all cases Electronic Flow Control is used for carrier gas control.

In many cases switching from one 1079 PTV mode to another involves changing the glass insert. A detailed stepwise procedure to carry out this task is given in the Maintenance section of this manual, on page 195. In most injection modes the insert is installed with a graphite ferrule. Use the special fixture supplied with the 1079 PTV accessory kit to install the ferrule correctly on the insert.

Isothermal Split Injection

The split mode is used when samples are relatively concentrated and for neat samples. This mode of injection involves rapid vaporization of the sample followed by sample splitting. Splitting involves directing a portion of the sample into the column while the remainder is vented. The split ratio is defined as the proportion of sample vented to the sample entering the column. With EFC carrier gas control this parameter can be set automatically in the 1079 PTV method.

Injector Insert	Install the fritted split insert (03-926119-46) or unpacked split insert (03-926119-45). See instructions in the Maintenance section on page 195 for changing the 1079 PTV insert.
Column Installation	7.5 cm from the bottom of the column nut at the base of the Injector
Injector Temperature	Isothermal 250 °C.
Column Temperature	50 °C initial for 0.1 min, ramp to 250 °C at 20 °C/min, hold 5 min.
Carrier Gas Control	Set the split mode to ON for the duration of the run in the 1079 PTV method section and set the split ratio to 100. The sample will be split upon injection and a representative portion representing 1/100th of the amount injected will enter the column. Note that the split ratio is a method specific parameter and should be set appropriately for individual analyses. In addition the Injector pressure or pressure ramp should be set to achieve the desired column flow rate.

Table 34: Split Injection Typical Conditions

Isothermal Splitless Injection

The classical splitless injection technique involves vaporizing the sample in a hot Injector and slow transfer to the column. The split state is OFF during the sampling period; therefore, all of the injected sample should enter the column. The initial column temperature is maintained for at least the sampling time to trap all sample components at the head of the column. At the end of the sampling period (typically 0.5 to 1.5 minutes) the split state is turned ON to vent any residual sample or solvent from the Injector.

The following table describes typical method parameters for an isothermal splitless injection.

Injector Insert	Install the standard 3.4 mm ID splitless insert (P/N 03-926119-45).
Column Position	7.5 cm from the bottom of the column nut at the base of the Injector.
Injector Temperature	Initial temperature isothermal 250 °C.
Column Temperature	50 °C initial for 1 min, ramp to 250 °C at 20 °C/min, hold 5 minutes.
Carrier gas control	Set the initial split mode to OFF and time program it to ON after 0.75 minutes. In this case the splitless sampling time is 0.75 minutes. The split ratio during the split ON period should be set to 50. Set the appropriate pressure or pressure ramp to achieve the desired column flow rate.

Table 35: Isothermal Splitless Injection Method Parameters

Temperature Ramped Splitless Injection

This is a similar technique to isothermal splitless except the sample is vaporized slowly, utilizing temperature programming. This controlled mode of vaporization reduces the risk of mass discrimination and thermal breakdown of sample components during the sampling period. The sample is deposited in the Injector as a liquid, therefore the initial Injector temperature must be close to the solvent boiling point and there must be a nearby surface for the sample to be retained on. For this mode of injection either a narrow bore (0.5 mm) insert or a glass wool packed insert is used. This facilitates efficient transfer of the sample to a surface from which it is then vaporized.

Note that the glass wool packet insert is not recommended for low levels of polar analytes.

The following are typical method parameters for a temperature ramped splitless injection. In this example hexane is used as the solvent which has a boiling point of 68 °C. Note that the sampling time and initial column hold time are 2 minutes to allow the Injector get to maximum temperature.

Injector Insert	Install either the narrow bore 0.5 mm ID insert (03-926119-49) or the glass wool packed 2 mm ID insert (03-926119-53) or the open 3.4 mm ID splitless insert (03-926119-45).
Column Position	7.5 cm from the bottom of the column nut at the base of the Injector.
Injector Temperature	Initial temperature 65 °C, hold for 0.1 minute, ramp to 250 °C at 150 °C/minute, hold 10 minutes.
Column Temperature	50 °C initial for 2 min, ramp to 250 °C at 20 °C/min, hold 5 minutes.
Carrier gas control	Set the initial split mode to OFF and time program it to ON after 2.00 minutes. In this case the splitless sampling time is 2.00 minutes. The split ratio during the ON period should be set to 50. Set the appropriate pressure or pressure ramp to achieve the desired column flow.

Table 36: Temperature Ramped Splitless Injection

On-column Injection

The wide-bore on-column mode uses a wider bore insert with a taper to allow insertion of a 0.53 mm ID capillary column inside the insert. A standard gauge GC syringe (26s) is used to deposit sample inside the column.
In case narrow bore columns are required, a retention gap is used as non-coated 0.53 mm ID pre-column.
This enables true on-column injection in a narrow bore column while using standard syringes.

The Injector is temperature ramped to minimize sample decomposition due to thermal effects or active sites.
The on-column technique involves some special setup of the 1079 PTV Injector.

In the on-column mode the column has to be sealed within the insert. The Injector nut, septum, septum support and insert are removed from the Injector. The column is pushed up through the Injector until it protrudes past the top of the Injector. The tapered on-column insert is then pushed onto the column to make a seal between the polyimide coating on the column and the glass surface. The insert is then lowered into the Injector and the septum support installed **without** a graphite ferrule. The septum support must not be screwed all the way down or the septum will not seal.

The following are typical conditions for carrying out an on-column injection. The Injector should be maintained at 10 - 20 °C below the solvent boiling point at injection. In this example the solvent is hexane which has a boiling point of 68 °C.

Injector Insert	Install the on-column insert (P/N 01-900109-07) for wide bore (0.53 mm ID) columns.
Seal/Position Column	Seal the column within the tapered insert.
Injector Temperature	Initial temperature 50 °C, hold for 0.1 minute, ramp to 250 °C at 150 °C/minute, hold 10 minutes.
Column Temperature	50 °C initial for 2 min, ramp to 250 °C at 20 °C/min, hold 5 minutes.
Carrier gas control	Split does not apply in on-column mode. The Splitless vent flow must be set to 20 mL/min or greater in Setup. Delete any splitter program and set initial state to off in the Injector screen.

Table 37: On-Column Injection

Large Volume Injection

The large volume injection technique is used where the absolute lowest level of detection is required. Up to 250 µL of sample may be introduced into the 1079 PTV Injector. The sample is injected at a very slow rate while the Injector temperature is set a few degrees below the solvent boiling point. In the example below hexane is used as the solvent which has a boiling point of 68 °C.

Using the large volume injection technique the Injector is maintained in the split ON state at the beginning of the run to vent most of the solvent. The sample components are trapped in the Injector insert so the same type of insert is used as for temperature ramped splitless injection. The split state is then programmed to OFF and the Injector temperature ramped to transfer the sample components to the head of the column. The following are typical conditions for a large volume injection.

Injector Insert	Install either the narrow bore 0.5 mm ID insert (03-926119-49) or the glass wool packed 2 mm ID insert (03-926119-53).
Column Position	7.5 cm from the bottom of the column nut at the base of the Injector.
Injector Temperature	Initial temperature 66 °C, hold for 1 minute, ramp to 250 °C at 150 °C/minute, hold 10 minutes.
Column Temperature	50 °C initial for 3.00 min, ramp to 250 °C at 20 °C/min, hold 5 min.
Carrier gas control	Set the initial split mode to ON, time program it to OFF after 1.00 minutes and then back to ON after 3.00 minutes. The split ratio should be set to 50 during the split ON periods.

Table 38: Large Volume Injection

Note that in all the above cases the parameters given are generic and will have to be optimized for specific applications. Particular care should be taken with the large volume mode of injection where the initial Injector temperature and timing of the split states have to be carefully selected.

Testing the 1079 PTV Injector Performance

The following procedure describes how to test the performance of the 1079 PTV Injector. This is best done with a test sample containing an appropriate set of components for the installed detector. The following table lists the series of test samples available for Varian GC.

Test Sample	*Part Number*	*Concentrations of Test Compounds*
TCD	82-005048-01	3.00 μg/μl of C_{14}, C_{15} and C_{16} in iso-octane.
ECD	82-005048-02	33.0 pg/μl of lindane and aldrin in iso-octane.
PFPD	82-005048-03	20.0 ng/μl of n-dodecanethiol, tributylphosphate, methyl parathion; 4000 ng/μl of n-pentadecane in iso-octane.
TSD	82-005048-04	2.00 ng/μl of azobenzene, methyl parathion; 4.00 ng/μl malathion and 4.00 μg/μl C_{17} in iso-octane.
FID	82-005048-07	30 ng/μl of C_{14}, C_{15} and C_{16} in iso-octane.
Note: *If the FID test sample is not available, the TCD test sample can be used if first diluted 100:1.*		

Table 39: 1079 PTV Test Samples

To run one of these test samples, use the chromatographic conditions listed previously for the injection technique you are currently using. The detector should be operated at the most sensitive range, e.g., 12 for FID and TSD, 0.05 for TCD, 10 for PFPD and 1 for ECD. The resultant chromatogram should approximate that shown in the detector section of this manual. Some chromatographic interpretation information is given in the troubleshooting section of this manual.

Maintenance

The 1079 PTV Injector that can be operated in several modes. These modes include split, splitless, on-column and large volume injection. Typically, to change from one mode of operation to another involves changing the Injector insert. The insert should be replaced on a routine basis. This is especially important when dirty samples are analyzed.

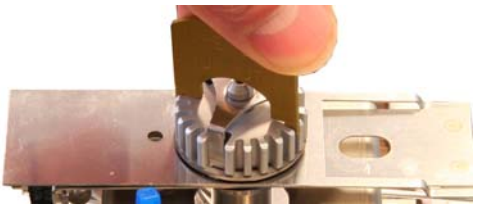
After prolonged use, the 1079 PTV Injector glass insert may need to be replaced with a new insert.

Tools Required

- Tweezers or septum pick (P/N 72-000084-00)
- Injector nut wrench (P/N 03-908423-00)
- Flat-blade screwdriver (short handle)
- Clean laboratory tissue
- Graphite ferrules (P/N 03-925342-01)
- Insert/ferrule positioning tool (P/N 03-925385-00)

Remove the Glass Insert

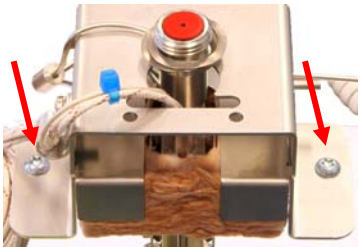
1. Use the Injector nut wrench to remove the Injector nut. Place the nut on a clean surface (e.g., clean tissue).



WARNING: BURN HAZARD

THE INJECTOR NUT MAY BE HOT. LOWER THE INJECTOR TEMPERATURE TO 50°C AND PERMIT THE INJECTOR NUT TO COOL BEFORE PROCEEDING.

2. Unscrew the 2 T-20 Torx screws holding the top of the Injector to the base. Carefully move this assembly to one side. The Septum purge line and carrier gas supply lines may hinder movement of the top piece somewhat.



CP-8400 AutoSampler, remove the Injector access plate by removing the 2 retaining screws and firmly lifting the plate. It may be hot if another Injector is also installed and powered. The plate may be a little tight, do not force it off. Lift straight up, a slight back and forth rocking may help remove it.

3. With tweezers or septum pick, remove the septum.

Replace the Injector septum each time the glass insert is replaced.

4. Use a clean flat-blade screwdriver to unscrew the septum support nut until it is loose.

5. Remove the septum support with the tweezers or septum pick.

Typically, when the septum support is removed, the insert and ferrule remain in the septum support. If the ferrule and insert are in the Injector body after the septum support nut is removed, use the tweezers to grasp the top of the insert and lift it from the Injector body.

6. Use a laboratory tissue to grasp the glass insert and remove it from the septum support nut.

7. To remove the graphite ferrule from the glass insert, use clean lab tissues to hold the graphite ferrule and the glass insert. Gently turn the glass insert while you pull off the graphite ferrule.

Replace the Glass Insert

1. Use the insert/ferrule positioning tool supplied in the 1079 PTV accessory kit to set the 5 mm graphite ferrule on the insert and in the septum support. See the pictures for an exploded view of the tool with septum support, insert ferrule, glass insert and tool as well as the correct position of the tool when setting the ferrule. The objective is to have the ferrule set with the bottom of the insert, flush with the bottom of the tool.

2. Position the tool as shown on a flat, clean surface. Use clean laboratory tissue on the surface. Tighten the septum support finger-tight. Holding the tool with a 5/8" wrench, give the septum support an extra 1/3 to 1/2 turn past finger-tight. Now unscrew the septum support which now has the ferrule and insert seated in it. If there is any graphite extruded past bottom of the septum support, cut it off with a blade or sharp knife. Carefully wipe off any graphite flakes which may adhere to the insert or septum support. Gripping the septum support unit with a piece of laboratory tissue, carefully put this unit in the 1079 PTV Injector and tighten the septum support 1/6-turn past finger-tight.

3. Use tweezers to place a new septum over the septum support.

If the septum has a Teflon face, place the Teflon face toward (down) the column.

4. Place the Injector nut on the Injector and tighten by hand until you feel some resistance, then tighten an extra 1/4 turn using the Injector nut wrench.

5. Condition the insert by setting the 1079 PTV Injector to the split mode and purging with carrier gas for 30 min at 300 °C.

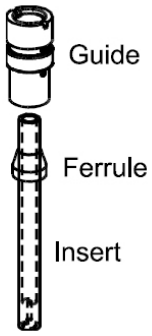
ChromatoProbe

How to Assemble the ChromatoProbe

Refer to the exploded view on the next page.
The ChromatoProbe assembly consists of the adapter, guide, ChromatoProbe, ChromatoProbe cap and the small and large O-rings. One small O-ring is placed on the ChromatoProbe shaft; the other is placed on the ChromatoProbe cap shaft. The large O-ring is placed in the groove in the bottom of the adapter which connects to the 1079 Injector body.
The storage stand may be used to hold and protect the ChromatoProbe or ChromatoProbe cap. The user may find the small holes in the top of the storage stand useful for holding microvials during filling or just prior to running an analysis.

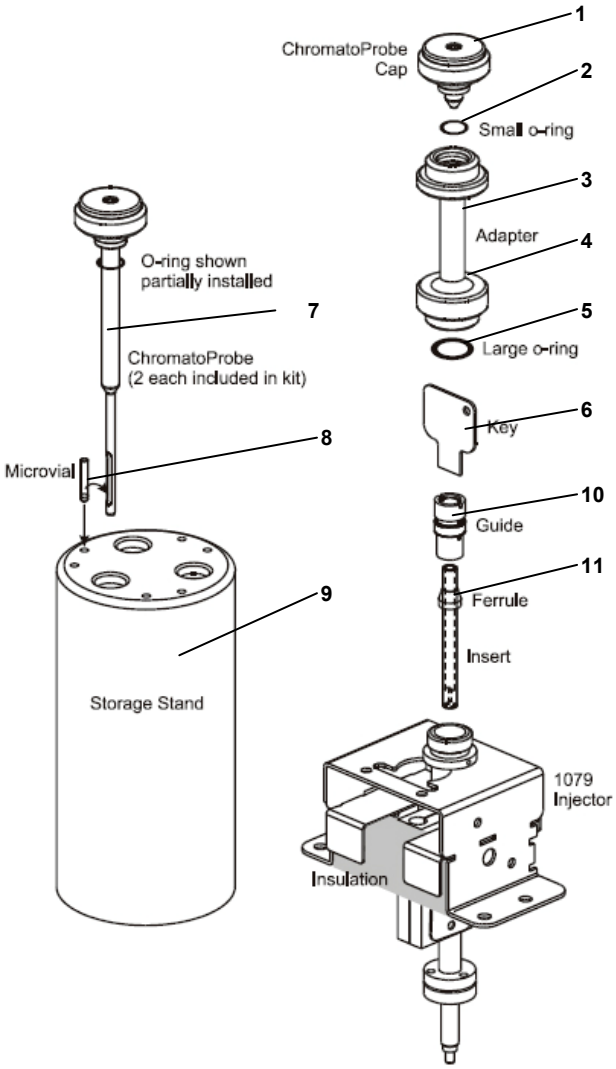
How to Install the ChromatoProbe

The ChromatoProbe is designed to be installed into the 1079 Injector body. Several parts of the Injector must be removed and replaced with the appropriate parts from the ChromatoProbe kit before the ChromatoProbe can be connected. Remove the Injector nut, septum, septum support (gold piece) and the Injector insert. The Injector nut, septum support and the Injector insert should be set aside for use when you use the Injector in the normal manner. Install the glass insert included in the ChromatoProbe kit into the Injector (3.4 mm split insert). This insert must be installed with the small orifice at the bottom, as shown in the picture. This position will allow the ChromatoProbe to slip into the insert during operation. Use the following procedure to set the insert, ferrule and the guide.



1. Place the 1/4 inch graphite ferrule over the open end of the insert so that the tapered end of the ferrule is up.
2. Place the insert and the ferrule into the guide so that the ferrule can fit snugly inside the guide.
3. Using the ferrule positioning tool (P/N 03-925385-00) included with the 1079 Injector, place the insert and guide into the tool and screw the guide clockwise with the key provided to tighten the ferrule around the insert. The insert is positioned correctly if the bottom of the insert is aligned with the bottom of the ferrule positioning tool. Refer to the maintenance section of the 1079 for removal of the glass insert on page 195.

Once the ferrule is positioned and tightened, the guide and insert can be placed into the 1079 Injector body. Screw the guide into the Injector body with the key and tighten snugly.
At this point the ChromatoProbe is ready to be attached onto the Injector. The adapter is attached to the Injector such that the portion containing the large O-ring is screwed onto the Injector. Tighten this fingertight to prevent any leaks. **DO NOT use a tool to tighten this connection**. Insert the ChromatoProbe cap into the top of the adapter and hand tighten to cap off the Injector when not using the ChromatoProbe.



ChromatoProbe Kit (03-925677-91)

1. ChromatoProbe Cap (03-925592-01)
2. Small O-ring, 0.239 (ID 03-930109-11)
3. Adapter (03-925589-01)
4. Large O-ring, 0.426 ID (03-930109-12)
5. Key (03-925690-01)
6. Guide (03-925582-01)
7. ChromatoProbe (03-925591-01)
8. Microvials (100/bottle) (03-925671-11)
9. Stand (03-925694-01)
10. Glass insert 03-918464-00
11. Graphite ferrule (5 mm) (2/pack) 03-925342-02

- Passivation Lacquer (03-925699-01)
- Tweezers (29-899510-00)
- Ferrule (0.4 mm) (10/pack) (28-694580-01)
- Ferrule, 2-hole (0.4 mm) (28-694559-01)
- Column nut (03-949551-00)

Figure 27: ChromatoProbe Assembly

NOTE: If it appears that there is an alignment problem while inserting the ChromatoProbe, follow this alternative method for installing the adapter:

1. Connect adapter to the Injector **without tightening it**.
2. Insert the ChromatoProbe through the adapter with a loose fit and then tighten the adapter to the Injector while the ChromatoProbe is inserted.
3. Finger tighten to prevent any leaks. **DO NOT use a tool to tighten this connection**.

A 2m x 0.10 mm fused silica column with 0.1 µm methyl silicone coating is provided for use with the ChromatoProbe. For a gas chromatograph containing 2 Injectors, it is possible to have the ChromatoProbe and a traditional column connected simultaneously. In this case, please refer to here on page 202. If only one Injector exists or the ChromatoProbe is installed alone, please continue as follows:

1. Remove any column that you have installed in the GC/MS and replace it with this 2 meter column.
2. Use the 0.4 mm single hole ferrule and the column nut provided in the kit.
3. The column should be inserted a distance of 7.5 cm into the Injector (standard for 1079 Injectors). The mass spectrometer side is set so that the column end is 1 mm past the end of the transfer line.
4. Set the head pressure to 10 psi and measure a flow out of the split vent of 50 mL/min. This will provide a flow rate of about 1 mL/min into the mass spectrometer and a split ratio of about 50:1.
5. Condition the Injector and the column at 300°C for 30 minutes. If an EFC is included, provide 1 mL/min column flow rate with split vent of 50 mL/min.

Using the ChromatoProbe

How to Perform Analysis with the ChromatoProbe

Liquids, solids and slurries, or even samples such as blood, urine, or milk can be analyzed using the ChromatoProbe.
Place the sample into a microvial provided with the kit. Solid or powder samples are easier to introduce to the microvial as a liquid solution with a standard syringe. This way is also cleaner and has smaller memory effects.
Alternatively, powders can be introduced into the microvial with the help of a Pasteur pipette. The microvial containing the sample is placed into the microvial holder position at the bottom of the ChromatoProbe shaft. Refer to the exploded view if this is unclear.
Remove the ChromatoProbe cap from the adapter which should be installed on top of the Injector and position the ChromatoProbe through the adapter, into the Injector.
Finger tighten the ChromatoProbe to seal the Injector. The sample is volatilized by raising the Injector port temperature.

The column can be hot and isothermal or temperature programmed to match the Injector. Since most analyses of solid samples do not require high sensitivity, the split vent should be opened during the entire analysis to only allow low nanogram amounts into the mass spectrometer.
Adjust the split flow to achieve a fast response, reduce thermal decomposition and to prevent the mass spectrometer from being overloaded and contaminated. A good starting value is about 50:1. If the split ratio is too high for a particular sample, rerun a fresh sample with a lower split ratio to get the desired sensitivity. In order to prevent overloading of the system, either load the smallest amount of solid possible or preferably dissolve the solid and load 1 µL of about 0.1% solution. Note Injector comment below.
Samples can be analyzed in any of the mass spectrometer modes of operation (EI, CI, MS/MS, CI-MS/MS). In general, use AGC target values of 5,000-10,000 to ensure a high spectral quality. A suggested temperature program for the 1079 PTV Injector and column oven is as follows.

INJECTOR:	120°C (or 20°C above the boiling point of the solvent if a solution is introduced) for 1 minute, then heat at 40°C/min to 300°C and hold for 4.5 min.
COLUMN OVEN:	140°C for 1 minute, then heat at 40°C/min to 300°C and hold for 5 min.

If liquids are to be analyzed, the user must empirically determine certain parameters to prevent sample from being inadvertently expelled out of the microvial during analysis, caused by sudden boiling (“bumping”) or spraying. These parameters include maximum fill level in the microvials; maximum initial temperature and heating rates of the Injector.

Analysis of Thermally Labile Samples

Thermally labile samples may decompose in the injection port. The hot metal surface of the ChromatoProbe can contribute to thermal decomposition resulting in improper spectra.
In order to eliminate this, Varian has introduced Silcosteel® coated ChromatoProbes.

Using the ChromatoProbe Along with Traditional GC/MS

For gas chromatographs containing 2 Injectors it is possible to have both the ChromatoProbe column and a traditional column connected simultaneously. This can be accomplished by employing a 2-hole ferrule and connecting both columns into the mass spectrometer. When the ChromatoProbe is not in use, replace the probe with the ChromatoProbe cap.
Heat the ChromatoProbe Injector to about 300°C with the split vent open to remove any contamination from previous samples. Now, reduce the ChromatoProbe Injector head pressure to 3-5 psi to minimize the helium flow into the mass spectrometer. The total flow of helium should remain between 1 and 1.5 mL/min to insure the best sensitivity and spectral quality. Other options include a “Y” connector for the 2 columns going into a single transfer line into the mass spectrometer. Be aware of the possible problems of surface activity when using this type of connection.

Using the ChromatoProbe as a Dirty Sample Inlet

The ChromatoProbe can be used as an effective tool for chromatographic analysis of dirty samples, without cleanup or extraction. This use of the ChromatoProbe is based on intra-Injector thermal extraction of the semivolatile compounds in the sample microvial, while non-volatile residue is retained in the microvial, which is disposed of after the analysis. For effective use of the ChromatoProbe with dirty samples, the following advice should be considered:

1. The sample microvial should be handled with *tweezers only*. Similarly, the microvial holder should not be touched with the hands to avoid fingerprints and dirt being included in the analysis.

2. Introduce into the microvial the smallest sample size that is easy to quantitatively transfer with a syringe. This is typically 1-3 microliter liquid samples.

3. Solid samples might be blended or dissolved for a more quantitative transfer, as well as for more efficient thermal extraction from the microvial.

4. The sample should be introduced at an Injector temperature about 20°C above the solvent boiling temperature, to enable fast but gentle solvent vaporization without sample splashing from the microvial. This temperature can be 120°C for water (urine) or 90°C for acetone blended fruit and vegetables.

5. The initial column temperature should be low enough to trap the extracted semivolatile compounds. For example, 50-80°C initial column temperature is desirable for pesticide analyses.

6. Solvent vaporization takes about one minute for each 3-4 microliter sample. The split vent can be opened during that time, with a split flow rate above 20 mL/min to speed up the solvent vaporization.

7. After approximately one minute solvent vaporization time, the Injector temperature should be fast programmed to 250°C (for pesticides) or the appropriate temperature required for the thermal extraction of the semivolatile compounds. The final temperature chosen is a compromise between higher temperature for more effective and faster thermal extraction and a lower temperature value that might be required to prevent thermal degradation of delicate compounds.

8. During the Injector heatup and thermal desorption time, the split valve must be closed and the carrier gas flow rate must be at least 4-5 mL/min for effective thermal extraction. At lower carrier gas flow rates a higher Injector temperature will be required.

9. The thermal desorption stage takes between 0.5-2 minutes. After that time, the Injector is cooled down, the carrier gas flow rate is programmed to the chromatographic optimal value and the GC oven program can begin in the usual way. Note that the final GC oven upper temperature and time can be significantly reduced since the less volatile compounds are retained in the microvial.

10. At the end of the analysis, dispose of the sample microvial. Do not re-use microvials. The microvial holder should only be removed from the adapter after both the GC oven and Injector are cool and a helium purge flow from the ChromatoProbe protects the column.

11. In addition to passivation of the microvial holder, analysis of thermally labile sample compounds is facilitated with a shorter microvial (up to 6 mm long). The standard 15 mm long microvials can be cut (like a column) to the desirable length. Handle the microvial carefully while cutting to avoid contamination.

12. The microvial volume is 30 microliters. Up to 20 microliters can be loaded and the solvent evaporated outside the GC prior to sample introduction.

13. Test your method for thermal vaporization efficiency, reproducibility and long term stability before beginning routine analysis.

Cleaning

Should the ChromatoProbe become contaminated with time, it can be cleaned by placing it in a solvent such as acetone and placed in a sonic bath. If it is severely contaminated a mild abrasive can be used to clean it.

Recommended Reading

1. Aviv Amirav and Shai Dagan, “A Direct Sample Introduction Device for Mass Spectrometry Studies and GC/MS Analysis”. European Mass Spectrometry, 3, 105-111 (1997).

2. Hongwu Jing and Aviv Amirav, “Pesticide Analysis with the Pulsed Flame Photometric Detector and a direct sample Introduction Device”. Analytical Chemistry, 69, 1426-1435 (1997).

3. Samuel B. Wainhaus, Shai Dagan, Mark L. Miller and Aviv Amirav, “Fast Drug Analysis in a Single Hair”. Submitted for publication.

THE 1041 ON-COLUMN INJECTOR

The 1041 On-Column Injector is designed for on-column sample injection onto 530 micron I.D. fused silica columns, 1/4 inch and 1/8 inch O.D. packed columns (glass or metal).
On-column injection provides complete sample transfer from Injector to column results in good quantitation.
The fused silica column or glass column extends all the way from the Injector septum to the detector, providing a clean inert system without cross-over's.
Quick and easy to switch from megabore to packed column or vise versa by changing an adaptor. The 1041 is supplied with capillary mounting hardware as standard. If you wish to operate the Injector in the packed column mode then a packed column adapter kit must be ordered (03-925588-91). This kit contains Injector and detector hardware to facilitate use of the Injector with 1/8" metal packed columns. In addition, adapter kits are available for 1/4" columns. Full instructions on converting the 1041 to packed column use are included with the packed column kit.

The 1041 On-Column Injector is available in manual pneumatics and electronic flow control (EFC) for different laboratory requirement.
Choose from EFC23 (constant flow control) and EFC24 (constant pressure control and constant total flow control) for different application requirement.

Automatic Start Switch

The automatic start switch is a spring loaded actuator that fits over and is aligned with the injection port of the 1041 universal Injector nut. The GC run is started when the actuator is depressed by the syringe barrel, or manually pressed at the moment of sample injection. The GC run can also be manually (only in local automation) started by pressing START .

WARNING:
BURN HAZARD

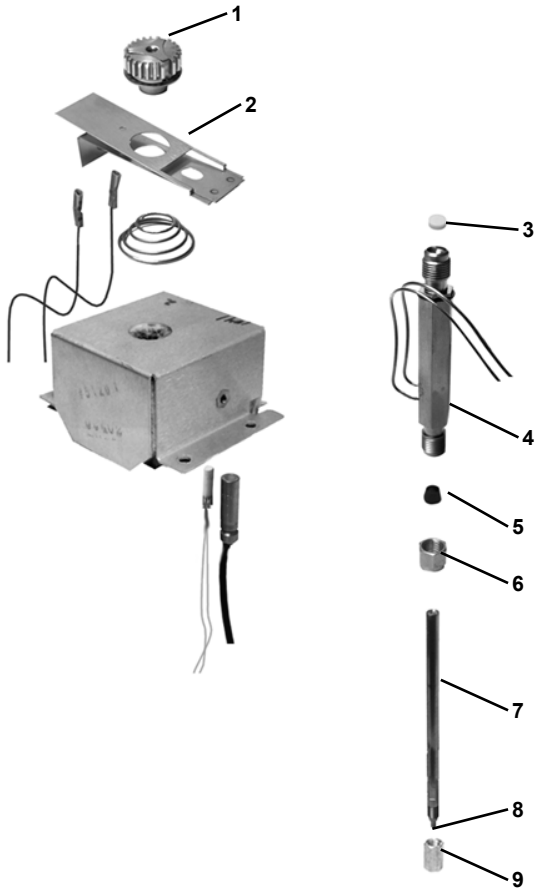
The Injector nut and automatic start switch assembly may be very hot during instrument operation and should not be touched with unprotected hands.

1041 Injector Insert

Click here to see the 1041 Injector Insert, on page 453.
Note that all 1041 Injector inserts are deactivated for maximum inertness.

Injector Assembly and Insert

A cross-sectional view of the 1041 On-Column Injector with insert and 530µm ID fused silica capillary column is shown below.



1. Injector Nut (390812700)
   Injector Nut Wrench (390842300)
2. Automatic Start Switch (390820601)
3. Septum, 9.5 mm
   BTO (lowest bleed, CR298705)
   Marathon (Autosampler, CR239188)
   Advanced Green 3 (general purpose, CR246124)
   Septum pick (7200008400)
4. Injector Body
   EFC23 type (392548201)
5. Injector Body Ferrule
   40% Graphite/60% Vespel (up to 400°C) (CR213400)
   Graphite (up to 450 °C) (CR211400)
6. Injector Body Nut (Stainless Steel, SWSS4021)
7. Injector Insert (Stainless Steel, 392543101)
8. Column Ferrule, see table below
9. Capillary Nut (394955100)

Figure 28: 1041 On-Column Injector with Insert

Column ID	*Teflon* *Max 250°C*	*Vespel* *Max 350°C*	*40% Graphite 60% Vespel Max 400°C*	*Graphite* *Max 450°C*	*SilTite* *Metal, GC/MS*
0.53 mm ID	CR214108	CR212108	CR213108	CR211108	SG073302

Table 40: 1041 Column Ferrules 1/16" x 0.8 mm

1041 Electronic Flow Control (EFC)

The Electronic Flow Control module used on a 1041 Injector can be type EFC23 or EFC24.

The EFC23 is used to control the 1041 Injector under flow control to program the column flow. EFC23 sets a required carrier flow into the Injector/Column system. A leak in the system would be indicated to the user by a drop in inlet pressure.

The EFC24 is used to control the 1041 Injector in combination with Headspace sampler and other devices that have a long carrier gas line.

Both EFC types are designed specifically for the 1041 Injector to support its various modes of operation. In simple terms it duplicates the behavior of the 1041 manual pneumatics system in that there is an inlet mass flow controller supplying carrier gas to the Injector and a pressure control valve downstream from the Injector which sets the Injector pressure. As Injector pressure determines the rate of carrier gas flow through the column, this pressure is monitored close to the point of injection.

The type EFC23 flow diagram on the next page shown is an indication of the control mechanism of this type of EFC module.

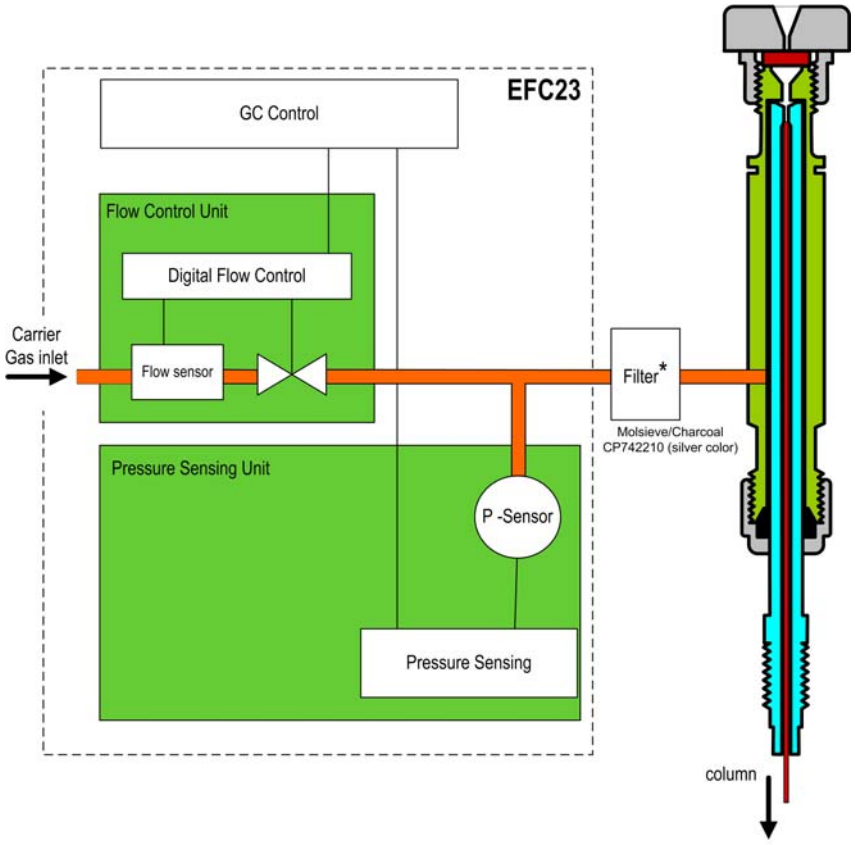


Figure 29: EFC23 and 1041 Flow Diagram

*See Pencil Filters on page 437

The type EFC24 flow diagram shown below is an indication of the control mechanism of this type of EFC module.

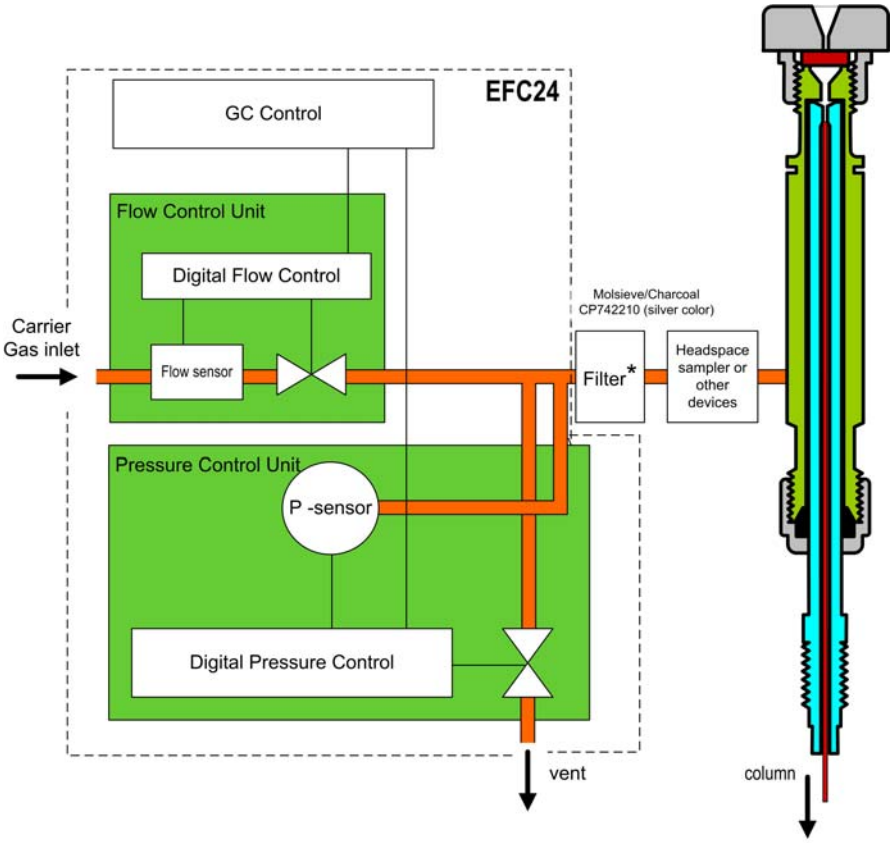


Figure 30: EFC24 and 1041 Flow Diagram

* See Pencil Filters on page 437

Column Installation

The Connect Capillary Column to Injector on page 15 procedure describes the installation (or reinstallation) of a capillary column in a Varian 450-GC equipped with a 1041 Injector. Each step of the procedure is described in detail.

Condition the Column

For conditioning the capillary column see Column Installation and Conditioning on page 23.

Column Installation in Detector

For connecting a capillary column into a detector, see Connect Capillary Column to Detector on page 24.

Setting 1041 Gas Flow Rates

The gas flow rates for the 1041 On-Column Injector can be set using manual pneumatics or Electronic Flow Control (EFC). Note that a positive flow through the column must be set before heating the column.

CAUTION

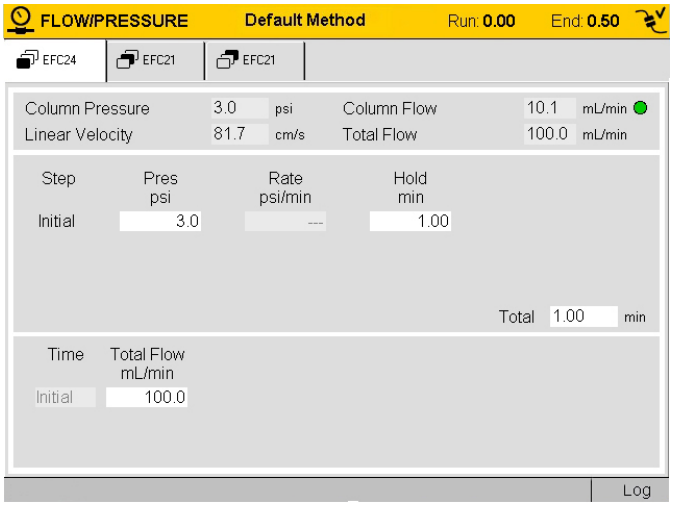
Do not heat the column oven above 50 °C without carrier gas flowing through the column. The column phase can be irreversibly damaged by exposure to oxygen at elevated temperatures. Allow 10 – 15 minutes to purge the column before heating.

EFC Pneumatics

Type EFC24 used with the 1041 or 1061 Injectors allows the user to set a constant column head pressure, build a pressure program, or set a constant column flow. With EFC23 equipped Injectors a flow range of 0 – 100 mL/min may be set from the 450-GC keyboard. A pressure program is typically used to maintain the column flow at a constant value while temperature programming the column oven. When Constant Flow Programming is enabled, the pressure program needed to maintain constant flow is derived whenever the method is loaded. The only user settable parameter with the 1041 type of EFC is the desired flow rate.

The 1041 is a pressure-controlled Injector; thus the column flow decreases with increasing column temperature if the pressure remains constant. EFC method parameters and status are accessed via the FLOW/PRESSURE key on the 450-GC keyboard.

The next screen is an example if a type EFC24 is used.



Flow Rates for Operation

Recommended flow rates differ, depending on the operating mode used. Refer to the appropriate paragraph for your operational mode.

Capillary Mode

When operating the Injector in the capillary mode, the following carrier gas flow rates are recommended:

Carrier Gas	*Column Flow Rate (Optimum)*	*Column Flow Rate (Typical)*
Nitrogen	1.5 mL/min	3 - 15 mL/min
Helium	2.4-4.0 mL/min	4 - 15 mL/min
Hydrogen	4.0-8.0 mL/min	8 - 20 mL/min

Table 41: Carrier Gas Flow Rates for 1041

Packed Mode

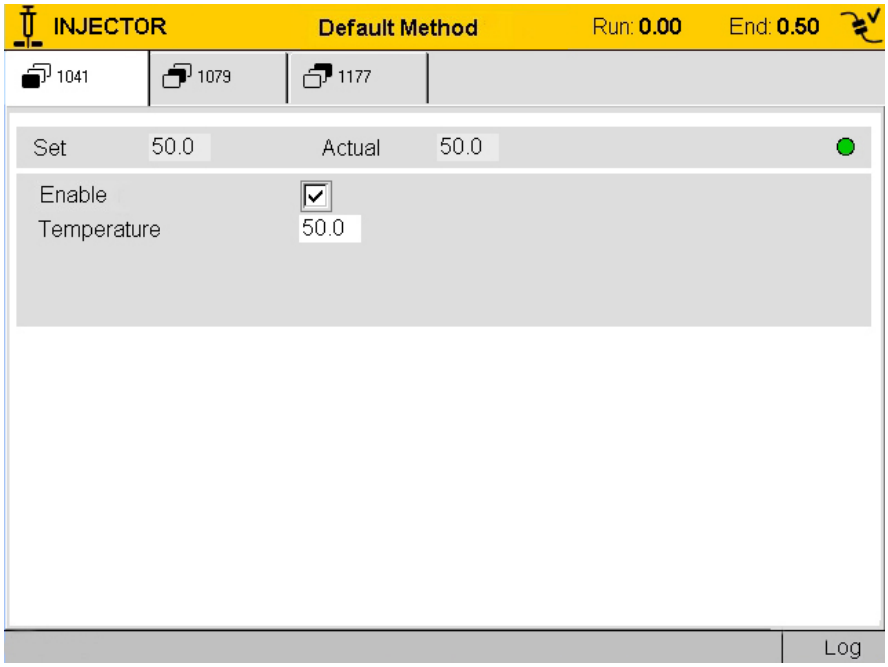
When operating in the packed column mode, adjust the carrier gas flow to applicable packed column flow rates (20 to 50 mL/min), depending on carrier gas type.

1041 Operation

This screen contains the specific operating information and procedures required for optimum performance of the 1041 on-column Injector. All installations for both the instrument and the Injector must be completed before continuing further.

1041 Injector Method

The 1041 Injector method component on the 450-GC keyboard contains one screen for setting the required Injector temperature and viewing the status information. The 1041 is an isothermal Injector and cannot be temperature programmed.



Testing the 1041 Injector

The most effective method of testing Injector performance is by running a test sample. The following procedure describes how to test the performance of the 1041 on-column Injector. This is best done with a test sample containing an appropriate set of components for the installed detector. The Table 61, on page 419 lists the series of test samples available for Varian GC detectors.

To run one of these test samples, use the chromatographic conditions listed below. The detector should be operated at its most sensitive range.

Example chromatograms can be found in the detector section on page 419 of this manual.

Injector temperature	250 °C
Column temperature	50 °C initial, ramp to 250 °C at 20 °C / minute and hold for 5 minutes
Injection Volume	1 µL

Table 42: 1041 Injector Chromatographic testing conditions

Observe the eluting peaks for symmetry, separation and elution time. Abnormally wide or skewed peaks, excessive elution times, abnormally small peaks and noisy or drifting baselines indicate faulty performance.

Maintenance

Hardware Replacement and Cleaning Procedures

Routine cleaning and maintenance of the 1041 On-Column Injector system includes septum replacement and column conditioning. Pressure testing and leak checking procedures are included to assure proper functioning of the system.

Septum Replacement

Septum replacement represents the major part of routine chromatographic maintenance. Septum damage from the needle penetrations can be avoided by injecting into the same hole and not using syringes with needles having burrs or bends at the tip which cut the septum.

WARNING: BURN HAZARD

The Injector nut and automatic start switch assembly may be very hot during instrument operation and should not be touched with unprotected hands. Allow sufficient time for the Injector nut and surrounding assemblies to cool before continuing with this procedure.

CAUTION

Handling a septum with bare fingers may result in column contamination. Use tweezers, finger cots, or gloves when installing a new septum.

1. Be sure the Injector nut is cool. Unscrew the Injector nut (use the Injector Nut wrench) and place on a clean, uncontaminated surface.
2. Using tweezers, remove the septum, taking care not to scratch the internal surfaces of the Injector.
3. Using tweezers, place a new high temperature septum in the Injector. If the septum is TFE or FEP coated put that side down.
4. Replace the Injector nut and tighten until resistance is felt, then tighten an extra 1/2 to full turn.

Needle/ Syringe Cleaning

For normal use, rinse the needle/syringe by slowly drawing up and quickly expelling solvent or the next sample to be used. Repeat the process several times.

When rinsing with the next sample, DO NOT expel sample back into the sample container

Leak Checking

Leak checking methods used with the 1041 On-Column Injector are presented in order of overall sensitivity and performance. When leak checking the Injector only, be sure to use the appropriate procedure.

1. Remove the test column and plug the Injector outlet with a 1/16" no-hole ferrule (P/N 28-694590-01). Remove the Injector nut, install a new septum, then replace Injector nut and turn nut clockwise until it comes to a stop.

2. Pressurize the Injector to 400 kPa (60 psig) with carrier gas. Turn off the carrier gas flow at the supply.

3. A pressure drop less than or equal to 3 kPa (0.5 psig) in 15 minutes is acceptable. Locate leaks with an appropriate leak detector.

Any changes in temperature while performing this test may result in false readings due to expansion/contraction of the gas with temperature.

CAUTION

Commercial soap type leak detection fluids should not be used at any point in a capillary system, since, if a leak is present, the fluid will penetrate and contaminate the system. Column performance will be degraded and a substantial period of time may be required to achieve a clean system.

4. An alternate way to leak check the Injector only is to have the column installed in the Injector end and the opposite column end sealed with a flame or appropriate fitting.

Syringe Leak Checking

In some instances, non-reproducible chromatographic responses can be attributed to a worn and/or leaky syringe. The syringe must be leak tight. To check the syringe, insert the needle in an Injector operating at 150-200 kPa (20 to 30 psig) with a no-hole ferrule. Place a drop of solvent at suspect leak locations and look for bubbles.

CAUTION

Commercial soap type leak detection fluids should not be used at any point in a capillary system, since, if a leak is present, the fluid will penetrate and contaminate the system. Column performance will be degraded and a substantial period of time may be required to achieve a clean system.

THE 1061 FLASH VAPORIZATION INJECTOR

The 1061 flash vaporization Injector is designed for use with wide-bore columns of 0.53 mm ID.
Used with packed glass liner with vaporization volume for dirty samples.
Glass liner is removable from the top. No need to disconnect the column.
The 1061 Injector can operate in 2 modes: the capillary column mode or the packed column mode.
Switching from one mode to another involves changing the Injector and detector column mounting hardware.
The 1061 is supplied with capillary mounting hardware as standard.
If you wish to operate the Injector in the packed column mode then a packed column adapter kit must be ordered (03-925588-92). This kit contains Injector and detector hardware to facilitate use of the Injector with 1/8″ metal packed columns.
Complete instructions are provided in this kit for installing and operating the Injector in the packed column mode.

Automatic Start Switch

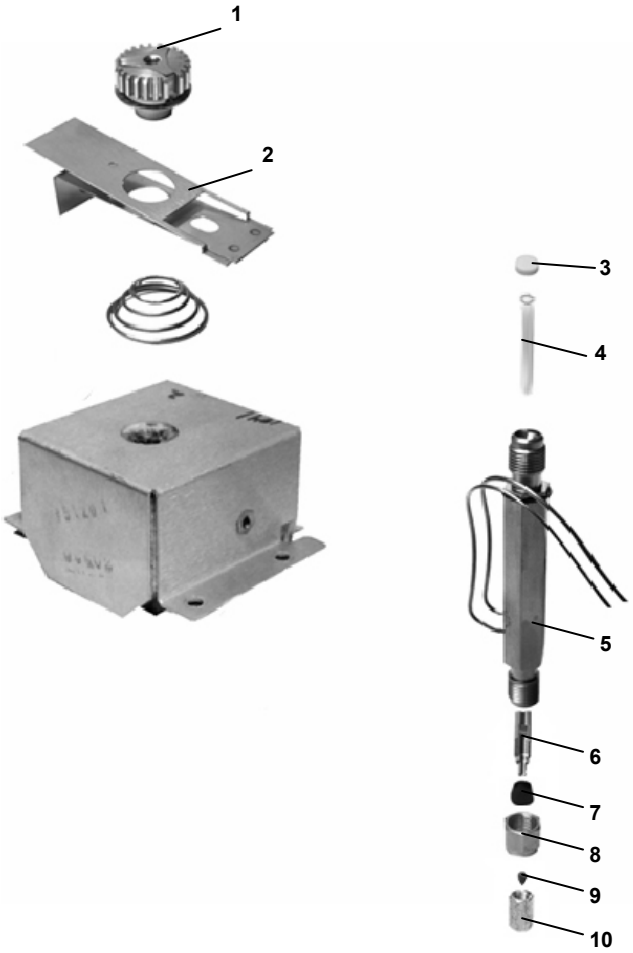
The automatic start switch is a spring loaded actuator that fits over and is aligned with the injection port of the 1061 Injector nut. The GC run is started when the actuator is depressed by the syringe barrel, or manually pressed at the moment of sample injection. The GC run can also be manually (only in local automation) started by pressing START .

WARNING:
BURN HAZARD

The Injector nut and automatic start switch assembly may be very hot during instrument operation and should not be touched with unprotected hands.

Injector Assembly and Insert

The 1061 Flash Injector with insert and 530µm ID capillary column



1 Injector Nut (392595501)
Injector Nut Wrench (390842300)
2 Automatic Start Switch (390820601)
3 Septum, 9.5 mm
BTO (lowest bleed, CR298705)
Marathon (Autosampler, CR239188)
Advanced Green 3 (general purpose, CR246124)
Septum pick (7200008400)
4 Glass Insert, default 392611943 More liners
5 Injector Body, EFC23 (392548301)
6 Column Guide (392558301)
7 Ferrule,
40% Graphite/60% Vespel (up to 400°C) (CR213400)
Graphite (up to 450 °C) (CR211400)
8 Injector body Nut (SWSS4021)
9 Column Ferrule, see table below
10 Bottom Nut (394955100)

Figure 31: 1061 Flash Vaporization Injector with Insert

Column ID	*Teflon* *Max 250°C*	*Vespel* *Max 350°C*	*40% Graphite 60% Vespel Max 400°C*	*Graphite* *Max 450°C*	*SilTite* *Metal, GC/MS*
0.53 mm ID	CR214108	CR212108	CR213108	CR211108	SG073302

Table 43: 1061 Column Ferrules 1/16" x 0.8 mm

1061 Electronic Flow Control (EFC)

The Electronic Flow Control module used on a 1061 Flash Injector can be type EFC23 or EFC24. The EFC23 is used to control the 1061 Injector under flow control to program the column flow. A leak in the system would be indicated to the user by a drop in inlet pressure. The EFC24 is used to control the 1061 Injector in combination with Headspace Sampler or other devices that have a large carrier gas line.

Both EFC types are designed specifically for the 1061 Injector to support its various modes of operation. In simple terms it duplicates the behavior of the 1061 manual pneumatics system in that there is an inlet mass flow controller supplying carrier gas to the Injector and a pressure control valve downstream from the Injector which sets the Injector pressure. As Injector pressure determines the rate of carrier gas flow through the column, this pressure is monitored close to the point of injection. The type EFC23 flow diagram shown below is an indication of the control mechanism of this type of EFC module.

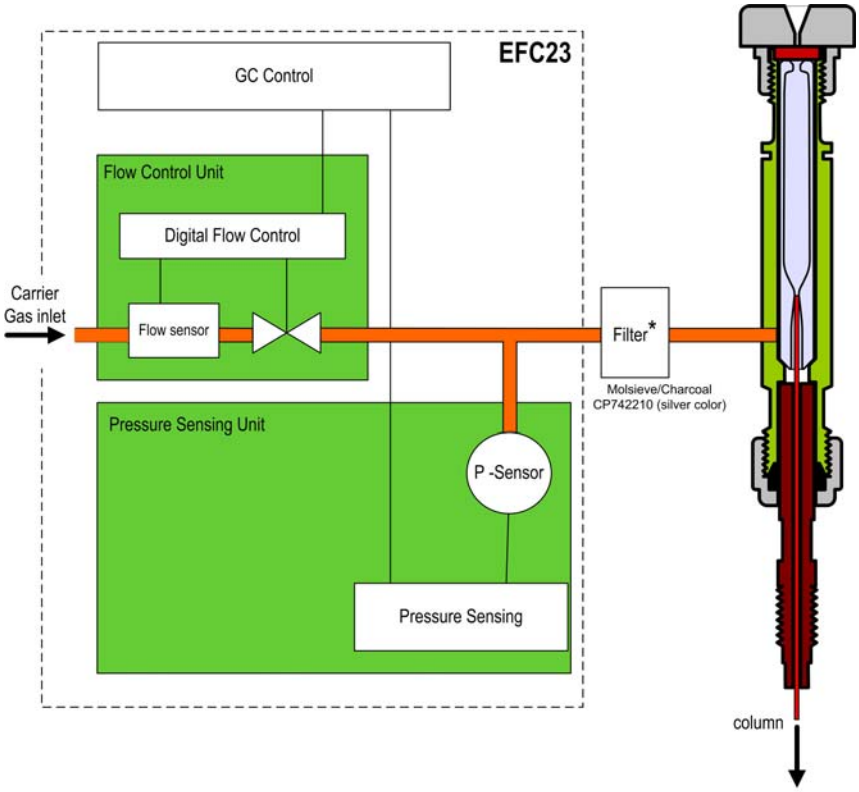


Figure 32: EFC23 and 1061 Flow Diagram

* See Pencil Filters on page 437

The type EFC24 flow diagram shown below is an indication of the control mechanism of this type of EFC module.

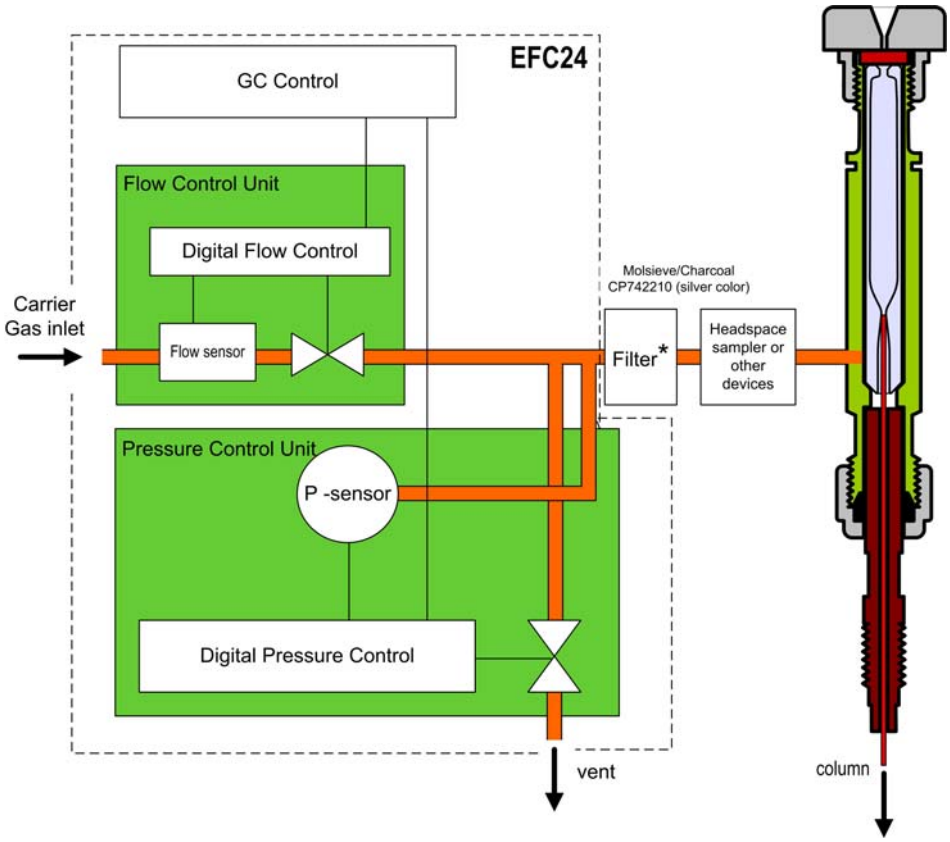


Figure 33: EFC24 and 1061 Flow Diagram

* See Pencil Filters on page 437

1061 Injector Insert

Click here to see the 1061 Injector Insert, on page 453.
All 1061 Injector inserts are deactivated for maximum inertness.

The 1061 Injector contains a glass insert with a tapered section on the bottom. This allows a wide bore capillary column seal with the insert. The sample is vaporized in the insert and then swept onto the column. The use of glass wool in the insert allows the analysis of samples containing non-volatile components. The non-volatile material is trapped on the glass wool while the volatile components vaporize onto the column. The packed column kit for the 1061 also contains a glass insert, in this case the packed column buts up against the bottom of the insert.

Column Installation

The following instructions apply to installing a 0.53 mm ID capillary column in the 1061 Injector. Note that the thin polymeric coating on fused silica columns will give some protection against breakage; however, fused silica columns are somewhat fragile and must be handled with care. Alternative is to use UltiMetal 0.53 mm wide-bore columns.

Column Installation

The Connect Capillary Column to Injector on page 15 procedure describes the installation of a capillary column in a Varian 450-GC equipped with a 1061 Flash Injector. Each step of the procedure is described in detail.

Condition the Column

For conditioning the capillary column see Column Installation and Conditioning on page 23.

Column Installation in Detector

For connecting a capillary column into a detector, see Connect Capillary Column to Detector on page 24.

Setting 1061 Gas Flow Rates

The gas flow rates for the 1061 Flash Injector can be set using manual pneumatics or Electronic Flow Control (EFC). Note that a positive flow through the column must be set before heating the column.

CAUTION

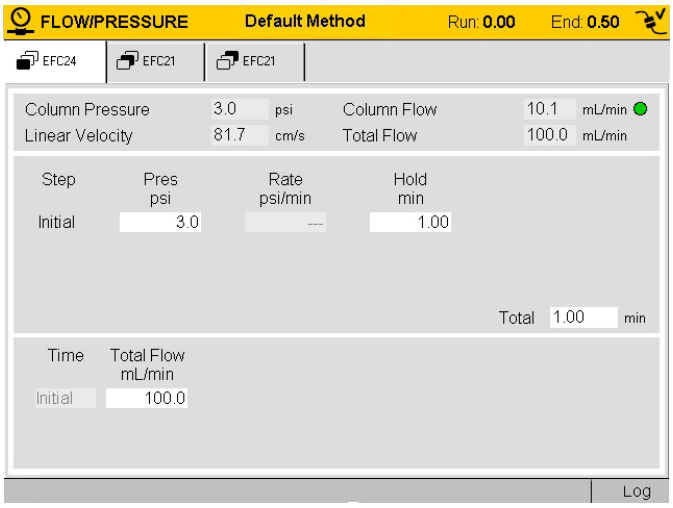
Do not heat the column oven above 50 °C without carrier gas flowing through the column. The column (stationary) phase can be irreversibly damaged by exposure to oxygen at elevated temperatures. Allow 10 – 15 minutes to purge the column before heating.

EFC Pneumatics

Type EFC24 used with the 1061 Injector allows the user to set a constant column head pressure, build a pressure program, or emulate a constant column flow when the column is temperature programmed. With EFC23 equipped Injectors a flow range of 0 – 100 mL/min may be set from the 450-GC keyboard. A pressure program is typically used to maintain the column flow at a constant value while temperature programming the column oven. When Constant Flow Programming is enabled, the pressure program needed to maintain constant flow is derived whenever the method is loaded.

The 1061 is a pressure-controlled Injector; thus the column flow decreases with increasing column temperature if the pressure remains constant. EFC method parameters and status are accessed via the FLOW/PRESSURE key on the 450-GC keyboard.

The next screen is an example if a type EFC24 method.



Flow Rates for Operation

Recommended flow rates differ, depending on the operating mode used. Refer to the appropriate paragraph for your operational mode.

Capillary Mode

When operating the Injector in the capillary mode, the following carrier gas flow rates are recommended:

Carrier Gas	*Column Flow Rate (Optimum)*	*Column Flow Rate (Typical)*
Nitrogen	1.5 mL/min	3 - 15 mL/min
Helium	2.4-4.0 mL/min	4 - 15 mL/min
Hydrogen	4.0-8.0 mL/min	8 - 20 mL/min

Table 44: Carrier Gas Flow Rates for 1061 Flash Injector

Adjust the make-up gas flow to give a total of 30 mL/min.

Packed Mode

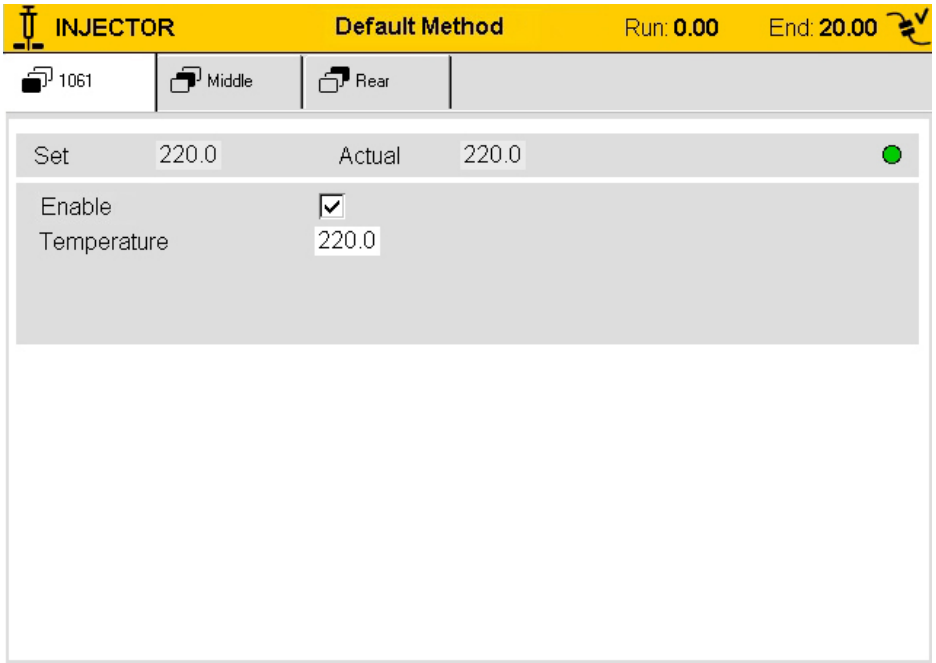
When operating in the packed column mode, adjust the carrier gas flow to applicable packed column flow rates (20 to 50 mL/min).

1061 Flash Injector Operation

This section contains the specific operating information and procedures required for optimum performance of the 1061 flash Injector. All installations for both the instrument and the Injector must be completed before continuing further.

1061 Flash Injector Method

The 1061 Injector method component on the 450-GC keyboard contains one screen for setting the required temperature for the Injector and viewing the status information. The 1061 Flash Injector is an isothermal Injector. The temperature of the 1061 Flash Injector must be optimized in order to have good sample evaporation in the liner.



Testing the 1061 Flash Injector

The most effective method of testing Injector performance is by running a test sample. The following procedure describes how to test the performance of the 1061 flash vaporization Injector. This is best done with a test sample containing an appropriate set of components for the installed detector. The Table 61, on page 419 lists the series of test samples available for Varian GC detectors.

To run one of these test samples, use the chromatographic conditions listed below. The detector should be operated at its most sensitive range.

Example chromatograms can be found in the detector section on page 419 of this manual.

Injector temperature	250 °C
Column temperature	50 °C initial, ramp to 250 °C at 20 °C/minute and hold for 5 minutes.
Injection Volume	1 µL

Table 45: 1061 Flash Injector Chromatographic Testing Conditions

Observe the eluting peaks for symmetry, separation and elution time. Abnormally wide or skewed peaks, excessive elution times, abnormally small peaks and noisy or drifting baselines indicate faulty performance.

MAINTENANCE

The 1061 is mechanically similar to the 1041 except that it uses a glass insert for flash vaporization injection. Follow the same maintenance procedures for the 1061 that are outlined above for the 1041, except in the case of changing or cleaning glass inserts.

Replacing the 1061 Glass Insert

After prolonged use, the 1061 glass insert may need to be removed for replaced with a new insert. Note that there are 2 different glass inserts, one for use with wide bore (0.53 mm ID) capillary columns and one for use with packed columns. These inserts are not interchangeable.

- Wide bore glass insert, Part Number: 03-926119-43.
- Packed column glass insert, Part Number: 03-926119-44.

See also #Injector_Parts_1061 on page 453.

Remove the Glass Insert

Follow these steps to remove the glass insert from the 1061 Injector.

1. Use the Injector nut wrench to remove the Injector nut. Place the nut on a clean surface (e.g., clean tissue).

WARNING:
BURN HAZARD

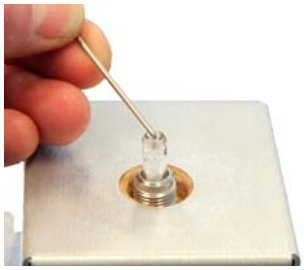
The Injector nut may be hot. Lower the Injector temperature to 50 °C and permit the Injector nut to cool before proceeding.

CP-8400 AutoSampler, remove the Injector access plate by removing the 2 retaining screws and firmly lifting the plate. It may be hot if another Injector is also installed and powered. The plate may be a little tight, do not force it off. Lift straight up, a slight back and forth rocking may help remove it.

2. With tweezers or a septum pick, lift the edge of the septum. Remove the septum.

Replace the Injector septum each time the glass insert is replace.

3. Use tweezers or a septum pick to grasp the glass insert and remove it from the Injector.



Replace the Glass Insert

Follow these steps to replace the glass insert in the 1061 Injector.

1. Pick up the new insert with tweezers and place it in the Injector carefully.
2. Use tweezers to place a new septum in the Injector.

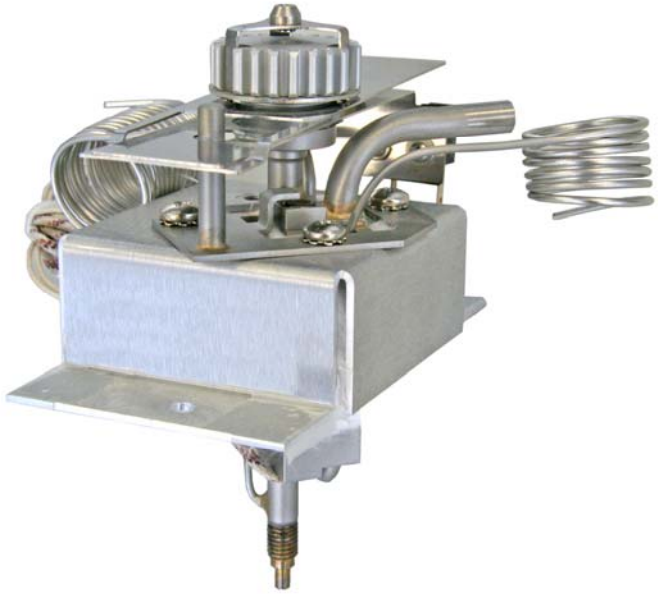
If the septum has a Teflon face, place the Teflon face toward (down) the column.

3. Place the Injector nut on the Injector and tighten by hand until you feel some resistance, then tighten an extra 1/2 to full turn using the Injector nut wrench.

After the Injector nut has been replaced check the head pressure (EFC status on display or manual pressure gauge on pneumatics panel) increases to its normal value. If this does not happen there is an indication of a leak.

4. Condition the insert by setting the 1061 Injector temperature to 300°C and allowing the system to condition for 30 minutes.

1093 SEPTUM-EQUIPPED PROGRAMMABLE INJECTOR (SPI)



The Varian 1093 Injector gives optimum performance for non-vaporizing (cold) injection into conventional fused silica capillary columns (0.32-0.53 mm ID columns).
It incorporates several unique design features to produce improved peak resolution, quantitation and versatility over other cold on-column Injectors.

The result is not only a capillary on-column injection system ideally suited for analyses that cannot be done with conventional split/splitless injection, but one that gives highly accurate and precise results with a wide variety of trace to semi-trace level samples.

The 1093 Injector can replace manually operated vaporizing capillary Injectors in many cases and may provide improved quantitation of labile and high molecular weight samples.

Temperature-programmed, cold injection: The Injector is held at a temperature 20 °C to 30 °C below the solvent boiling point during injection and then is rapidly temperature programmed up to the final column temperature.

The oven is started at the same temperature as the Injector. Conventional temperature programmed analysis is carried out in the GC column.

Frequently, good results may be obtained and the run time may be reduced by injecting into a cold Injector but with the column at 5°C above the solvent boiling point. In this case, the column is held at the initial temperature until the Injector is at its final temperature.

Cold injection reduces or eliminates many of the problems of conventional vaporizing split/splitless injection, such as molecular weight discrimination (loss of high and low boiling substances), thermal decomposition of the sample and sample adsorption onto hot, active Injector surfaces. These problems are largely eliminated in cold injection because the sample is directly introduced into the column as a liquid. In the split/splitless Injector, the sample is first vaporized and then allowed to pass into the column, where it may be condensed back into its liquid state.

The 1093 Injector may also be used as an isothermal, vaporizing Injector. However, best performance is achieved using the temperature-programmed mode. The system consists of three major components: a glass insert, the heated Injector body and a standard syringe. Maximum syringe needle diameter is 0.019" (0.49 mm) or 26 gauge.

All heated parts and all parts in direct contact with the carrier gas stream are constructed of stainless steel, borosilicate glass, fused silica, silicone rubber (septum) and Inconel X750.

The Injector body is mounted on an insulated enclosure. The Injector heating rate is adjustable to allow optimization for thermally sensitive samples. A purge exit is provided for bake-out and septum purge. The internal dead volume of the Injector body has been minimized.

In operation, the fused silica column is inserted up into the Injector body from the column oven and seated in the glass insert. The column is sealed to the Injector with conventional polyimide/graphite or graphite ferrules. The syringe needle enters the glass insert from the top. After the syringe is withdrawn, the entire injection zone is rapidly heated, driving the sample into the column where the peaks are sharpened by the solvent effect and cold trapping. Significant peak broadening occurs (up to 2X) in Injectors without this rapid heating step, especially when larger samples and high-boiling samples are used.

Of the 2 internal parts in the Injector body, the septum support and glass insert require periodic removal for cleaning. A biweekly check is recommended during regular use. The need for cleaning will vary with different samples and the glass insert is easily replaced in case of non-removable deposits or breakage.

The 1093 Septum-Equipped Programmable Injector (SPI) is factory-installed or available as a field upgrade (must be installed by trained personnel).

Automatic Start Switch

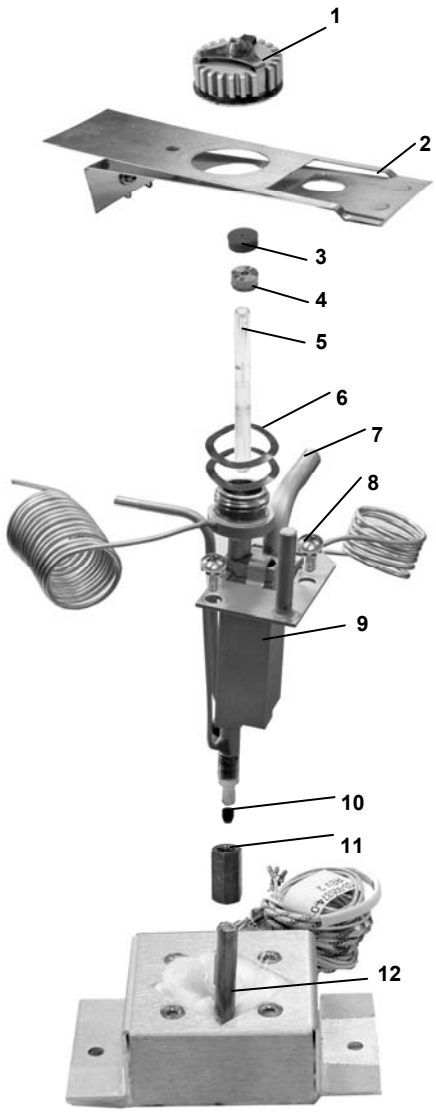
The automatic start switch is a spring loaded actuator that fits over and is aligned with the injection port of the 1061 Injector nut. The GC run is started when the actuator is depressed by the syringe barrel, or manually pressed at the moment of sample injection. The GC run can also be manually (only in local automation) started by pressing START .

WARNING:
BURN HAZARD

The Injector nut and automatic start switch assembly may be very hot during instrument operation and should not be touched with unprotected hands.

Injector Assembly and Insert

The 1093 Septum-Equipped Programmable Injector (SPI).



1. Injector Nut, (3949666601)
   Injector Nut Wrench, (390842300)
2. Automatic Start Switch (390820601)
3. Septum, 7/16”
   BTO CenterGuide™, (CR298717)
   Marathon (Autosampler, CR239287)
   Advanced Green 3 CenterGuide™ , CR246225)
   Septum pick (7200008400)
4. Septum Support, (391821100)
5. Glass Insert, default SG092034 More liners
   High performance (190010906)
   On-Column (190010907)
   Packed (392611950)
6. Wave spring washer 2x, (1492000501)
7. Coolant exit, Frit Restrictor (3600004601)
   Plug, 1/8” tube (2824707101)
8. Screws 3x, (391866306)
9. Injector Body
10. Ferrule,
    40% Graphite/60% Vespel (up to 400°C), CR213400
    Graphite (up to 450 °C), CR211400
11. Bottom Nut brass (394955100)
    Stainless Steel (CP743117)
12. Heater/probe 120 Vac, (391833400)
    230 Vac, (391833401)

Figure 34: 1093 Injector Assembly

1093 Injector Parts

Click here to see the 1093 Injector parts on page 454.

1093 Electronic Flow Control (EFC)

The Electronic Flow Control module used on a 1093 Injector can be type EFC23 or EFC24. The EFC23 is used to control the 1093 Injector under flow control to program the column flow. A leak in the system would be indicated to the user by a drop in inlet pressure. The EFC24 is used to control the 1093 Injector in combination with Headspace Sampler or other devices that have a large carrier gas line.

Both EFC types are designed specifically for the 1093 Injector to support its various modes of operation. In simple terms it duplicates the behavior of the 1093 manual pneumatics system in that there is an inlet mass flow controller supplying carrier gas to the Injector and a pressure control valve downstream from the Injector which sets the Injector pressure. As Injector pressure determines the rate of carrier gas flow through the column, this pressure is monitored close to the point of injection. The type EFC23 flow diagram shown below is an indication of the control mechanism of this type of EFC module.

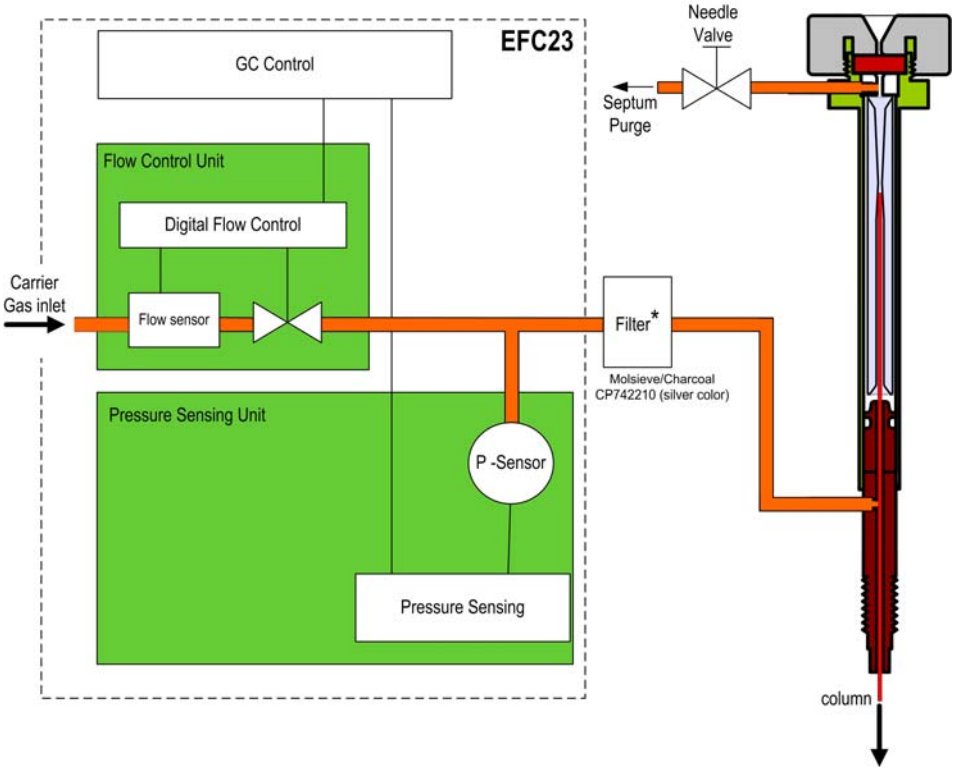


Figure 35: EFC23 and 1093 Flow Diagram

* See Pencil Filters on page 437

The type EFC24 flow diagram shown below is an indication of the control mechanism of this type of EFC module.

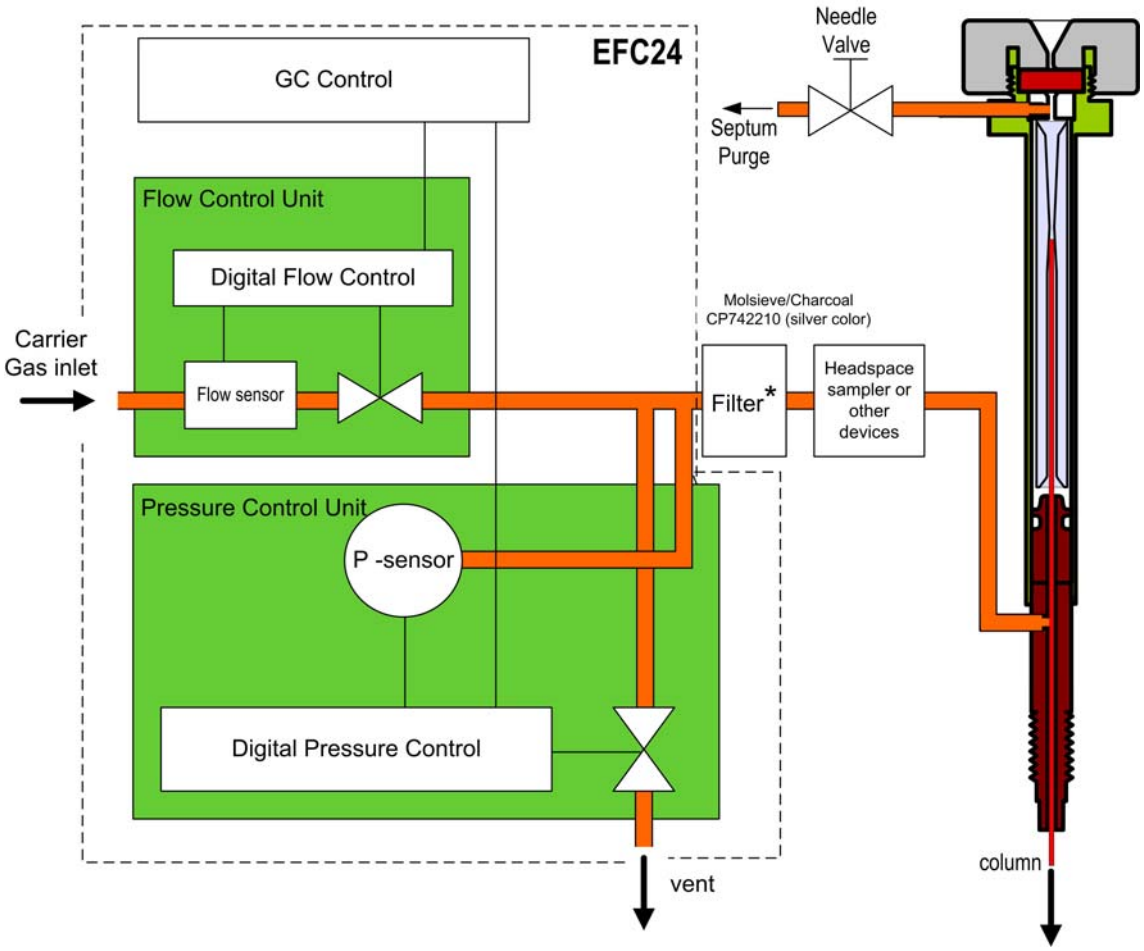
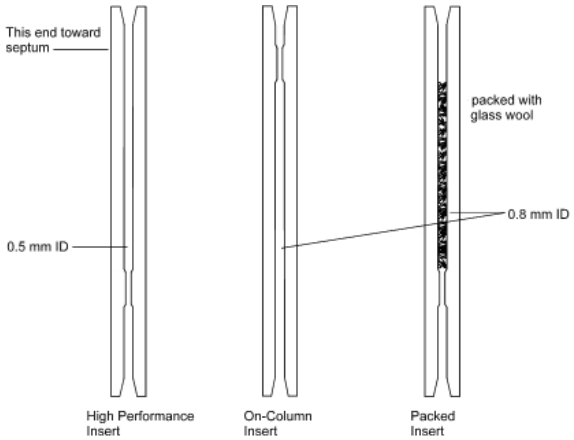


Figure 36: EFC24 and 1093 Flow Diagram

* See Pencil Filters on page 437

Glass Insert

Different glass inserts are available for use in the 1093 Injector.
Click here to see all inserts of the 1093 Injector parts on page 454
The standard insert is the high performance (P/N 190010906).



The high-performance insert (P/N: 190010906) is used when the presence of glass wool is unacceptable. The small internal diameter of this insert provides efficient transfer of the liquid sample from the syringe. If inserts with larger internal diameters are used without any packing, then sample transfer from the syringe may not be quantitative.
The on-column insert (P/N: 190010907) is used for on-column injection into large bore (0.53 mm ID) columns.
The glass wool packed insert (P/N: 392611950) provides efficient transfer of the liquid sample from the syringe and traps any contaminants present in the sample.

Syringe

The required syringe needle length is 2.00" (excluding the hub) for both manual injections and used in the CP-8400 and 8410 AutoSamplers.

Mounting the Column

During operation, the fused silica capillary column hangs on the capillary column holder in the column oven.
Hang the coiled column on the capillary column holder, then prepare the column end and complete column connections to both the Injector and detector fittings, as detailed in the following paragraphs.

Column Installation

The Connect Capillary Column to Injector on page 15 procedure describes the installation (or reinstallation) of a capillary column in a Varian 450-GC equipped with a Varian 1093 Injector. Each step of the procedure is described in detail.

Condition the Column

For conditioning the capillary column see Column Installation and Conditioning on page 23.

Column Installation in Detector

For connecting a capillary column into a detector, see Connect Capillary Column to Detector on page 24.

Connecting CO_2 to the 1093 Injector

The recommended tank for use with the 1093 Injector is a size 1A cylinder containing approximately 60 pounds of “bone dry” liquid CO_2. The tank should be equipped with a liquid eductor tube. Special fittings provided in the Start-Up Kit (P/N 03-918335-90) for the Injector are required for connection to the CO_2 tank. Refer to figure below.

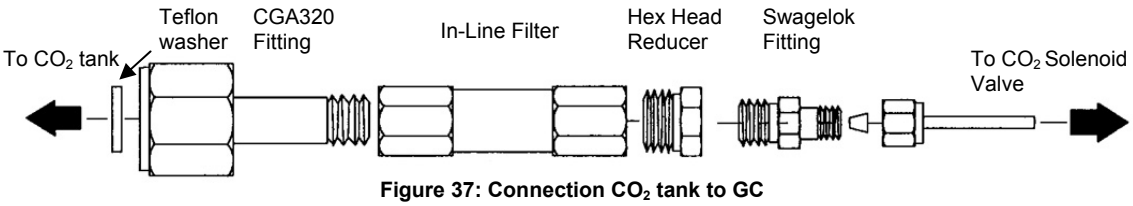


Figure 37: Connection CO_2 tank to GC

Approximately 100 chromatographic runs can be made per tank of CO_2 when the Injector is cooled to -50°C for each run. Cooling to higher temperatures will consume less coolant per run. The rate of use of CO_2 is dependent on the operational conditions, thus your actual use may vary from this estimate.

1. Use 1/8" copper or stainless steel tubing for the connection between the CO_2 solenoid valve and the CO_2 tank. **NOTE: If stainless steel tubing is used, change all fittings to stainless steel.**

2. Connect one end of the 1/8" Swagelok fitting to the GC and the other end of the tubing to the in-line filter and CGA 320 adapter. All hardware is supplied in the Start-Up Kit.

3. The CGA 320 fitting will be connected to the CO_2 tank. A Teflon washer is fitted in the nut that will connect to the tank and it must be inspected. Check that the washer is in good condition. If it is not, replace that washer and then connect the CGA 320 fitting to the tank. **NOTE: Do not over tighten the CGA 320 fitting. A high torque is not required, as the Teflon washer is the seal.**

Connecting LN2 to the 1093 Injector

When connecting LN_2 to the 1093 Injector, Neoprene insulation tubing (P/N 24-000467-00) must be installed over the entire length of tubing from the solenoid valve to the tank, to prevent ice build up and condensation. Consumption of LN_2 is approximately 0.5 to 1.0 pounds per run when the Injector is cooled to -50°C for each run. Cooling to higher temperatures will consume less coolant per run. The rate of use of LN_2 is dependent on the operational conditions, thus your actual use may vary from this estimate.

Full tank pressure is directed to the GC and the tank regulator should be less than 50 psi for stable operation.

WARNING

LN2 tanks that are designed for pressures higher than 50 psi must not be used with in-line regulators, shut-offs, or restrictors due to high pressure build-up. In all cases, never limit the tank's built-in venting system.

Compressed air at 40 psig may be used to cool the Injector using the LN_2 option; however, it is not recommended due to the low efficiency of cooling with air. If operation above 50°C is acceptable, then air may give satisfactory cooling rates.

Setting 1093 Gas Flow Rates

The gas flow rates for the 1093 Injector can be set using manual pneumatics or Electronic Flow Control. Note that a positive flow through the column must be set before heating the column.

CAUTION

Do not heat the column oven above 50 °C without carrier gas flowing through the column. The column phase can be irreversibly damaged.

EFC Pneumatics

The Electronic Flow Control module used on a 1093 Injector can be type EFC23 or EFC24.

The EFC23 is used to control the 1093 Injector under flow control to program the column flow. EFC23 sets a required carrier flow into the Injector/Column system. A leak in the system would be indicated to the user by a drop in inlet pressure.

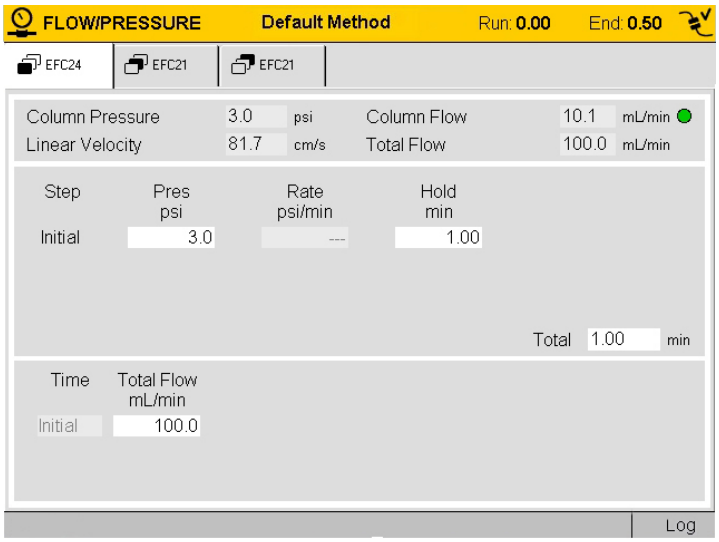
The EFC24 is used to control the 1093 Injector in combination with Headspace sampler and other devices that have a long carrier gas line.

Both EFC types are designed specifically for the 1093 Injector to support its various modes of operation. In simple terms it duplicates the behavior of the 1093 manual pneumatics system in that there is an inlet mass flow controller supplying carrier gas to the Injector and a pressure control valve downstream from the Injector which sets the Injector pressure. As Injector pressure determines the rate of carrier gas flow through the column, this pressure is monitored close to the point of injection.

Type EFC23 or EFC24 used with the 1093 Injector allows the user to set a constant column head pressure, build a pressure program, or set a constant column flow. A pressure program is typically used to maintain the column flow at a constant value while temperature programming the column oven. When Constant Flow Programming is enabled, the pressure program needed to maintain constant flow is derived whenever the method is loaded.

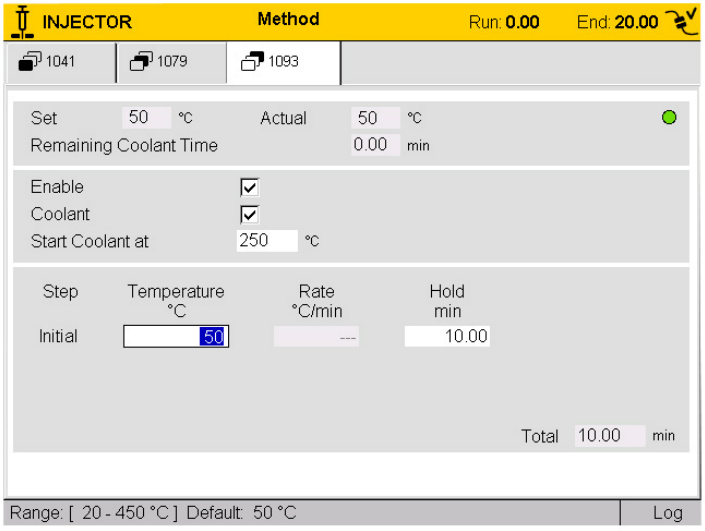
The 1093 is a pressure-controlled Injector; thus the column flow decreases with increasing column temperature if the pressure remains constant. EFC method parameters and status are accessible via the FLOW/PRESSURE key on the 450-GC keyboard.

The next screen is an example if a type EFC24 used.



1093 Injector Operation

This screen contains the specific operating information and procedures required for optimum performance of the 1093 Injector. All installations for both the instrument and the Injector must be completed before continuing further.
The 1093 Injector method component on the 450-GC keyboard contains one screen for setting the required temperature, Injection rate and hold time can be time programmed for the Injector.



Testing the 1093 Injector Performance

The following procedure describes how to test the performance of the 1093 Injector. This is best done with a test sample containing an appropriate set of components for the installed detector. The following table lists the series of test samples available for 450-GC.

Test Sample	*Part Number*	*Concentrations of Test Compounds*
TCD	82-005048-01	3.00 μg/μL of C_{14}, C_{15} and C_{16} in iso-octane.
ECD	82-005048-02	33.0 pg/μL of lindane and aldrin in iso-octane.
PFPD	82-005048-03	20.0 ng/μL of n-dodecanethiol, tributylphosphate, methyl parathion; 4000 ng/μL of n-pentadecane in iso-octane.
TSD	82-005048-04	2.00 ng/μL of azobenzene, methyl parathion; 4.00 ng/μL malathion and 4.00 μg/μL C_{17} in iso-octane.
FID	82-005048-07	30 ng/μL of C_{14}, C_{15} and C_{16} in iso-octane.
Note: *If the FID test sample is not available, the TCD test sample can be used if first diluted 100:1.*		

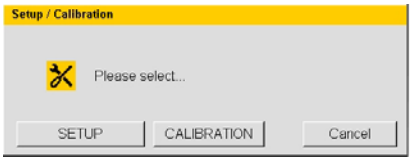
Table 46: Detector Test Samples

To run one of these test samples, use the chromatographic conditions listed previously for the injection technique you are currently using. The detector should be operated at the most sensitive range, e.g., 12 for FID and TSD, 0.05 for TCD, 10 for PFPD and 1 for ECD. The resultant chromatogram on page 419 should approximate that shown in the detector section of this manual.

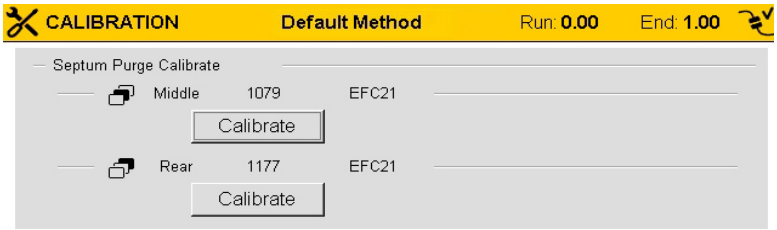
Observe the eluting peaks for symmetry, separation and retention time. Abnormally wide or skewed peaks, excessive retention times, abnormally small peaks and noisy or drifting baselines indicate faulty performance.
When the instrument is installed a definitive indication of instrument performance can be obtained by running a test chromatogram for the detector(s) that will be used with the Injector. This initial test chromatogram should be retained as a standard of comparison for later checking the instrument if change in detector sensitivity is suspected.

Septum Purge Calibration

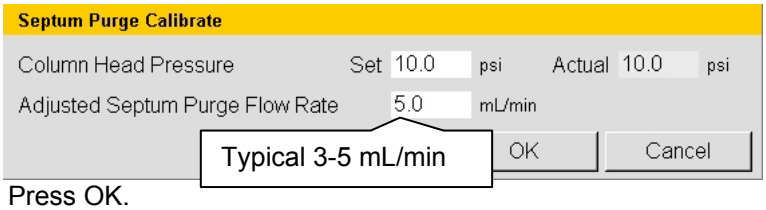
The 450-GC is equipped with a septum purge for the Model 1093 Injector and is controlled by a manual needle valve. This is located behind the front cover of the GC.
The manual needle valve can offer advantages over fixed restriction types as it can be adjusted to suit the more demanding applications. For the accurate display of total column flow and velocity it does require calibration when columns of different length and diameter are installed.
With type EFC23/24, the septum purge calibration routine should be carried out when the instrument is first set up or a new column is installed or when a significantly different pressure point is chosen. Press the SETUP key and click on Calibration.



The Calibration page will appear, choose Calibrate for the correct Injector (front, middle or rear).



Enter the desired Column Head pressure. After a few seconds measure the septum purge flow rate and adjust the septum purge valve (located behind the column oven door) to the desired flow, enter this value (typical 3-5 mL/min) in the Adjusted Septum Purge Flow Rate field.



Press OK.

Maintenance

Before maintaining the 1093 Injector please read the common Injector/septum maintenance information in the section Maintenance, on page 432.

Septum Replacement

Septum replacement represents the major part of routine chromatographic maintenance. Septum damage from the needle penetrations can be avoided by injecting into the same hole and not using syringes with needles having burrs or bends at the tip which cut the septum.

Handling a septum with bare fingers may result in column contamination. Use tweezers when installing a new septum.

Follow steps 1 through 7 below for replacing the septum in the Injector. Be sure to cool all heated zones to less than 80°C before opening the septum nut and decrease the column head pressure to less than 2 psig. If pressure is too high, the septum support and glass insert may pop up, rather than remain in their correct positions.

Removal of Glass Insert

Use care when removing or replacing the glass inserts for the 1093 Injector.

WARNING: BURN HAZARD

The Injector nut may be hot. Lower the Injector temperature to 50 °C and permit the Injector nut to cool before proceeding.

Removal

To avoid contaminating the capillary system, ALWAYS use tweezers or the extraction tool to handle the Injector internal parts.

1. Use the Injector nut wrench to remove the Injector nut and place on a clean, uncontaminated surface, such as a fresh cleaning tissue.

2. Remove the Injector switch and the 2 wave spring washers.

3. Using tweezers or the extraction tool (P/N: 7200008400) supplied in the Accessory Kit, remove the septum, septum support and glass insert. Place these parts on a clean, uncontaminated surface.

4. If the glass insert does not pop up when the septum support is removed, it may be necessary to push up on the column from the column oven. The polyimide on polymer-coated fused silica columns often forms a very tight seal to the glass insert. This seal must be broken before the insert can be removed.

Re-assemble

Install a new glass insert by following steps 5 through 8.

To avoid contaminating the capillary system, ALWAYS use tweezers or the extraction tool to handle the Injector internal parts.

5. Place the insert into the Injector body.

6. Install the septum support over the insert. The line on the septum support **must not be visible!**

7. Using tweezers, place a new high temperature septum over the septum support. If a Teflon-coated septum is used, the Teflon side must face down.

8. Place the Injector switch and the 2 wave spring washers and nut on the Injector and tighten until resistance is felt, then tighten an extra 1/4-turn.

Leak Checking

Leak checking methods used to leak check the 1093 Injector are presented in order of overall sensitivity and preference.

Leak Checking the Injector Only

1. Remove the (test) column and plug the Injector outlet with a 1/16" no-hole ferrule (P/N: 28-649503-00). Turn Injector nut clockwise until it comes to a stop. Turn off the septum purge.

2. Pressurize the Injector to 30 psig with carrier gas. Turn off the carrier gas flow at the ON-OFF valve.

3. The pressure should hold for 30 minutes, as shown on the status screen. A pressure drop less than or equal to 1 psig per minute at 30 psig is acceptable. Locate leaks with an appropriate leak detector.

Commercial soap type leak detection fluids should not be used at any point in a capillary system, since, if a leak is present, the fluid will penetrate and contaminate the system. Column performance will be degraded and a substantial period of time may be required to achieve a clean system.

4. An alternate way to leak check the Injector only is to have the column installed in the Injector end and the opposite column end sealed.

Gas Leak Checking

A convenient and non-contaminating way to leak test fittings and connections after installation or hardware replacement procedures is to direct a small jet of gas (butane from a disposable lighter is recommended) at the point to be tested, then use the detector, at maximum sensitivity, to detect leakage of gas into the system. Use normal column flow, a cool oven and an operational detector. If a peak is detected in excess of the magnitude given in table below, repair the leak.

Detector	***Gas***	***Attenuation, Range***	***Maximum Peak Size***
FID	Butane	16×10^{-12}	30%
ECD	Dichloromethane	2 x 1	50%
PFPD, Sulfur Mode	Natural gas containing Methanethiol	2×10^{-10}	50%
TSD	Dichloromethane	2×10^{-12}	2%

Table 47: Detectable Gas Leak Checking

Visual Leak Checking

The least preferred method of leak checking is to place a drop of pure solvent (isopropyl alcohol is recommended) on the suspected fitting or connection and look for bubbles.

Commercial soap type leak detection fluids should not be used at any point in a capillary system, since, if a leak is present, the fluid will penetrate and contaminate the system. Column performance will be degraded and a substantial period of time may be required to achieve a clean system.

Syringe Leak Checking

In some instances, non-reproducible chromatographic responses can be attributed to a worn and/or leaky syringe. The syringe must be leak tight. To check the syringe, insert the needle in an Injector operating at 20 to 30 psi (150-200 kPa) with a no-hole ferrule. Place a drop of solvent at suspect leak locations and look for bubbles while moving the syringe plunger up and down.

Injector and Column Conditioning

Initial bake out of a factory-installed Injector or test column is not necessary. If bake out becomes necessary due to contamination from handling, etc., follow the procedures or recommendations in the following paragraphs.

Baking Out the Injector

It may be necessary to bake out the Injector if it has become contaminated during the re-assembly process, i.e. if the internal parts were placed on dirty surfaces or touched with bare hands. Then, both the Injector and column should be conditioned.

To prevent contamination and overheating of the fused silica column, the column must NOT be connected to the Injector during the Injector bake-out procedure.

1. Disconnect column from Injector.
2. Fully open the septum purge.
3. Install a no-hole ferrule in the capillary column nut. Thread the nut up onto the assembly and tighten.

4. Set flow between 100 and 200 mL/min. Measure flow at the purge line exit, then remove the no-hole ferrule from the column nut.

5. Set the Injector temperature to 450°C.

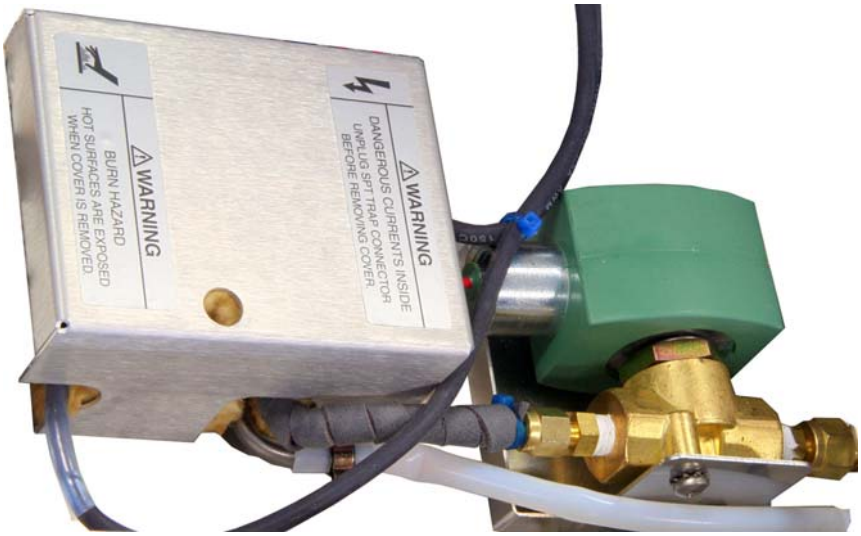
6. Bake out the Injector for not less than 4 hours, to a maximum of 16 hours at a time.

7. Cool the Injector to 50°C and then install the capillary column, on page 15.

8. Replace the septum.

9. Readjust the septum purge to the desired flow rate.

SAMPLE PRECONCENTRATION TRAP (SPT)



Description

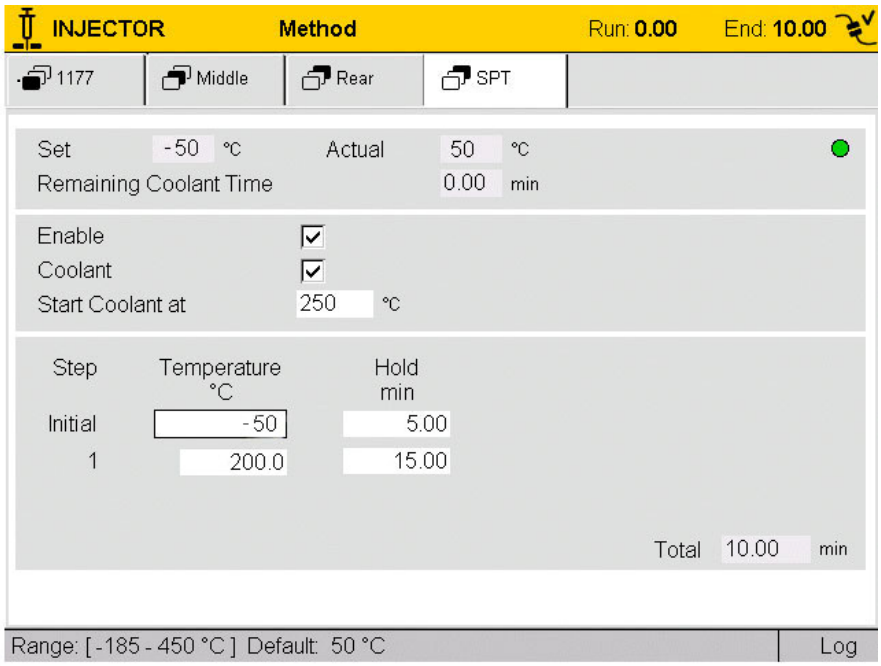
The SPT is a concentrating system for the determination of trace level volatile organic components in air and gaseous matrices. Integrated into the top of the GC, its design provides the following features:

- Fast heating rates.
- Very low cryogenic consumption.
- Short and long trap length options.
- Liquid carbon dioxide (LCO_2), liquid nitrogen (LN_2) cryogenic cooling.

Trap heating rates of up to 40 °C/sec provide fast injection from the trap to the capillary column. This helps to ensure good chromatographic performance without the need of an isolation valve, even for low boiling analytes.
Fast injection also allows higher initial column oven temperatures, thereby reducing or eliminating the need for column oven cryogenics. Cryogenic usage for trap cooling is minimized because of the compact design of the SPT and the specially designed algorithm that controls the cooldown and temperature of the SPT.

SPT temperature zone

The SPT temperature zone is independently controlled by a heated zone of the 450-GC for setting the trapping temperature and up to 2 desorption programs. Because of the trap's fast heating rates, there is no rate parameter setting for the desorption programs.



Each SPT option includes a SPT valve oven, installed either for operation with 2 gas switching valves or with one valve and one Injector. The SPT valve oven is controlled by the 450-GC heated zone and provides a constant temperature environment for the trap ends and tubing connecting them to an SPT valve. The SPT is assigned to heated zone 3 only.

The multi-valve oven may be installed on the 450-GC for SPT systems which require more than 2 valves.
See building an SPT method on page 263 for more details.

Two trap sizes available

Two trap sizes and various packing materials are available:

- The short trap is generally used for cryogenic trapping on glass beads using LN_2 or for highly adsorbing trap materials.
- The long trap length is suitable for trapping with adsorbents such as Tenax or charcoal-based materials at ambient or near ambient trapping temperatures where LCO_2 cryogen may be used.

The following are nominal measurements for the 2 trap sizes:

Trap Size	*Active Bed Length*	*Total Length*
Short	6.8 cm	17.02 cm
Long	29.2 cm	39.47 cm

Using the SPT

Trapping Methodology

There are fundamentally 2 methods of preconcentrating volatile organics: cryogenic trapping and adsorbent trapping. Cryogenic trapping takes place on an inert material, such as glass beads, at a very low temperature. For example, hazardous air pollutants with boiling points down to -30°C can be preconcentrated on glass beads at -160°C using LN_2.

The other trapping method uses adsorbents, such as Tenax, charcoal or other carbon-based materials, porous polymers, or coated column packings, alone or in various combinations at near ambient trapping temperatures. For example, the same pollutants mentioned above can be trapped on a Tenax/Charcoal, Carbopack B, or Vocarb 4000 (Supelco) packed trap at ambient temperatures.

Trapping Media

Several trapping media were mentioned above. In general, the least active medium that will still provide adequate capacity (retention volume) for the components should be used. This will keep the desorption temperature at a minimum and allow faster release of the analytes from the trap to the column.

Trap Dimensions

Once the trapping material is chosen, the trap size (long or short) should be selected based on the volume of the sample to be passed through it and the trapping temperature (all SPT traps are made from 1/8” o.d., type 316 stainless steel tubing). Most traps should be able to accommodate samples of approximately 400 milliliters at flow rates of about 40 mL/min in order to obtain the detection levels required.

Trap Temperature

The temperatures required for cryogenic trapping on glass beads are easy to determine. As noted above, a trap temperature of -160°C has been used for air toxics. When preconcentrating even lower boiling analytes, such as the C_2-hydrocarbons, trap temperatures as low as -180°C are necessary. When using adsorbents, the trapping temperature is determined by the breakthrough volume of the sample. Lower activity adsorbents require lower temperatures.

Trapping and Desorption

The flow rate during trapping is generally from 10 to 40 mL/min. Trapping is always more efficient at lower flow rates. Flow rate during desorption depends on the analytical system to which the analytes are passed. Desorption flow rates of 5 mL/min or greater are compatible with 0.53 mm i.d. columns and usually produce narrow peaks for early eluting components. When the capillary column i.d. does not allow this high flow, fast desorption flow rates in combination with inlet splitters may be used.

To improve peak shape and quantitation in cases where narrow bore column systems are in use but detectivity requirements prohibit the use of split inlet systems, column oven cryogenics will be required. This allows refocussing of early eluting components.

Electrical System

The SPT option provides independent temperature control of 2 parts of the trap assembly:

1. Heating and cooling of the trap.

2. Heating of the trap ends.

Trap temperature programming is accomplished in 450-GC heated zone 3, controlling heating and cryogen cooling of the trap. The SPT trap is mounted on the SPT valve oven, which maintains the SPT trap ends at a constant temperature. The oven is powered by one of the other remaining heated zones.

Extremely fast heating of the trap is made possible by utilizing the wall of the trap tubing as an electrical heating element. This eliminates the extraneous thermal mass associated with conventional heater cartridges. To match the electrical resistance characteristics of the trap tubing, a specific electrical voltage and current are provided by the SPT transformer (mounted in the rear of the GC). To provide the required voltages, the SPT trap harness connectors for the short and long trap are different. Although low voltages (less than 8 volts AC) are used to heat the trap, the SPT transformer is designed to deliver very high currents.

WARNING

The trap ends carry dangerous currents. Do not put any conducting material, such as wrenches, screwdrivers, or metal tubing, across the trap ends. Do not wear metallic jewelry while working near the trap. Variations in line voltage supplied to the GC will have an effect on the heating rate while the trap is desorbing.

Circuit breaker

The circuit breaker reset button is located above the SPT transformer on the rear panel of the GC. This pop-out button protects the SPT from abnormal power usage by interrupting the electrical connection to the trap. Wait at least 5 minutes after the circuit breaker is tripped before resetting it.

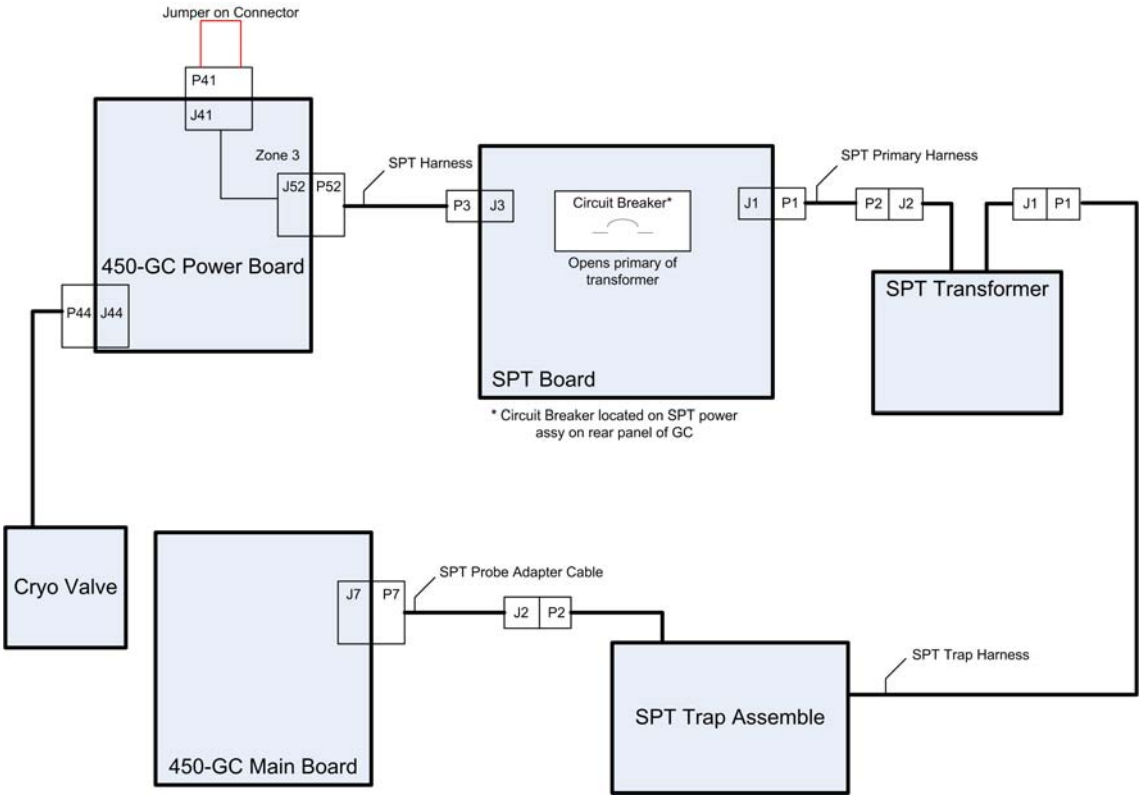


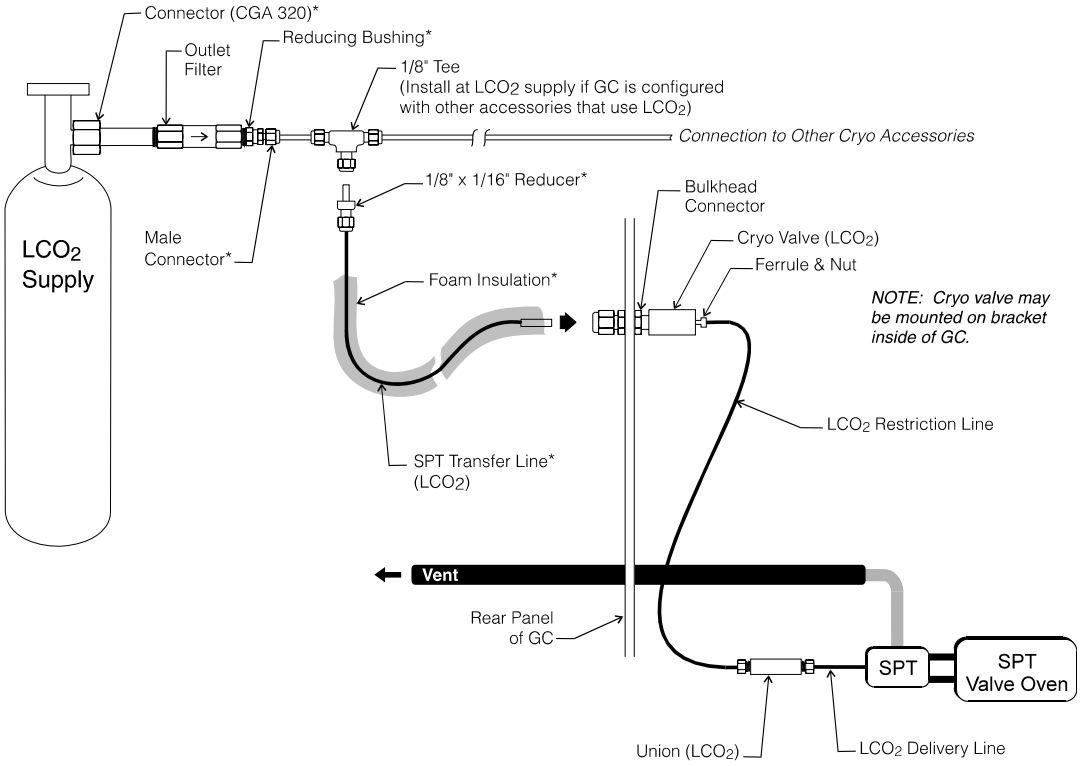
Figure 38: SPT Electrical Diagram

LCO_2 Option

System Description

The SPT-LCO_2 option allows trapping temperatures as low as -60°C. This system is very efficient and consumes a minimum amount of LCO_2.

Because of low coolant consumption, conditions that affect thermal efficiency of the coolant transfer system are very important to consider.



*Included with SPT Accessory Kit (LCO_2 Option)

Figure 39: LCO_2 Coolant Flow Diagram

Theory of Operation

As LCO_2 expands into the gas phase in the SPT cryo Enclosure, the latent heat of vaporization provides cooling power. The coolant flow rate is regulated by varying the pulse rate of the cryo solenoid valve.

The latent heat of vaporization drops rapidly as the temperature increases to the CO_2 critical point of 31°C. In order for the SPT to have adequate cooling power, LCO_2 should be delivered to the cryo valve at 27°C or cooler.

Characteristics of LCO_2 as a Coolant

The coolant for this option is liquid phase carbon dioxide (LCO_2) supplied from a pressurized cylinder[a]. At pressure, this cylinder contains both liquid and gas phases coexisting at equilibrium. The cylinder pressure is then equal to the saturation pressure and is dependent on the supply temperature. As the cylinder temperature rises, the cylinder pressure rises. Thus if a constant temperature is provided a constant pressure is maintained. This is true until the liquid phase CO_2 is depleted. For this reason it is advisable to keep the cylinder at a fairly “full” condition. Do not let it drop more than 60% of its weight.

Inadequate Cooling

To insure reliable cooling power and trapping temperature stability, the CO_2 must be delivered as liquid phase CO_2. When cooling power is lost, the CO_2 is often being delivered as gas phase. This may be caused by 2 different conditions.

1. CO_2 supply is too low. There is insufficient CO_2 remaining in the cylinder to build supply pressure to the saturation pressure.

2. The CO_2 has vaporized in the transfer line because the temperature of the transfer line is higher then that of the supply. The supply temperature must be equal to or warmer than the transfer line.

Locating the LCO_2 supply cylinder near the GC in a temperature controlled room is ideal.

[a] The critical point of CO_2 is 31 °C

Transfer Line

The SPT option comes with a 3 meter long, small diameter, low volume transfer line. The supply cylinder must be located then, less than 3 meter from the GC.

The low volume feature of the transfer line helps this option tolerate situations where the GC and transfer line are slightly warmer than the supply cylinder. Keep the transfer line cool and stable by insulating it with foam insulation provided in the Accessory Kit.

Recommended Supply Conditions

Supply	High pressure cylinder with siphon tube.
Supply Conditions	Temperature 15°C to 27°C (740 psia to 975 psia)
Outlet Filter	15 micron particulate filter (accessory kit) installed at the cylinder connection.
Transfer Line (from LCO_2 cylinder to the GC)	A 3 meter long, 1/16” dia. Stainless steel tube is included with this option. Install as a dedicated line from supply to SPT. Insulate with foam pipe insulation.

LN_2 Option

Description

The SPT - LN_2 option allows trapping temperatures as low as -190°C. For best utilization of the high performance features of the SPT, it is recommended that the transfer line from the LN_2 source to the GC be very efficient. The LN_2 transfer line should have the following characteristics:

- Minimal length.
- Should not have excessive restriction to flow.
- Low thermal mass.
- Well insulated.

In addition, the transfer line should not be routed near heat sources that could reduce the cooling efficiency, such as the column oven exhaust fan.

If the SPT system is at ambient temperature (e.g., 23°C) the time required for initial cooling of the SPT from 50 to -190°C might be longer (approximately 5-10 minutes) than subsequent cooling cycles.

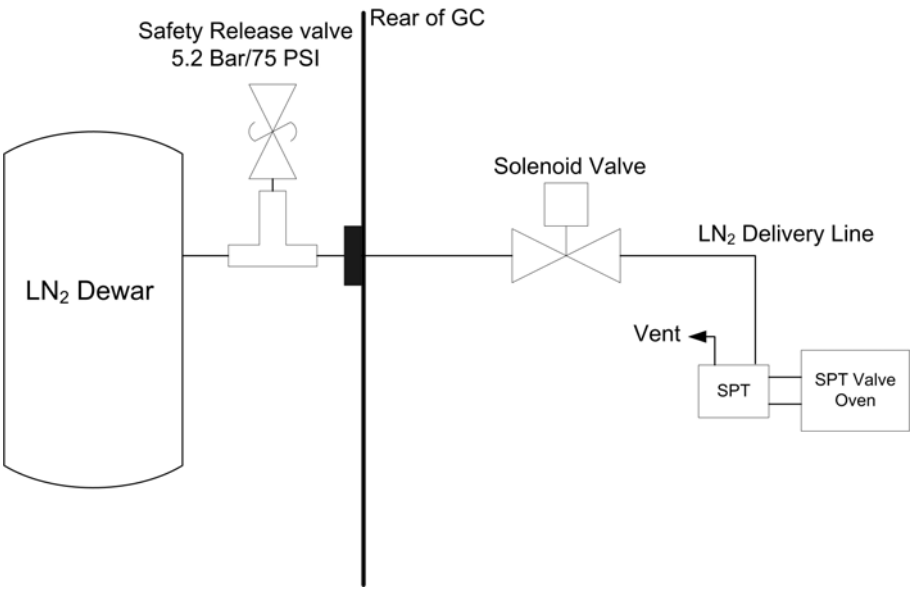


Figure 40: LN_2 Coolant Flow Diagram

Recommended Supply Conditions

WARNING

Due to high pressure build-up, LN2 tanks that are designed for pressures higher than 50 psi must not be used with in-line regulators, shut-offs, or restrictors. In all cases, never limit the tank's built-in venting system.

Supply	LN_2 tank configured for liquid delivery.
Supply Pressure	19 to 25 psig
Transfer Line (from LN_2 tank to GC).	1 to 3 meters length of (0.190" i.d.) ¼" o.d. copper tubing, insulated with foam insulation tubing.

Recommended temperature ranges

Parameter	***450-GC Range***	***Recommended***
Trapping Temperature	-60 to 450 °C -185 to 450	LCO2 - 60 to 50 °C LN2 - 185 to – 40 °C
Desorption Temperature Program 1	-185 or -60 to 450[a] °C	50 to 420°C
Desorption Temperature Program 2	-185 or -60 to 450[b] °C	50 to 420 °C
SPT Temperature Limit	-60 to 450 °C -185 to 450 °C	--
SPT Valve Oven	--	150 to 250 °C

Table 48: SPT recommended operating ranges

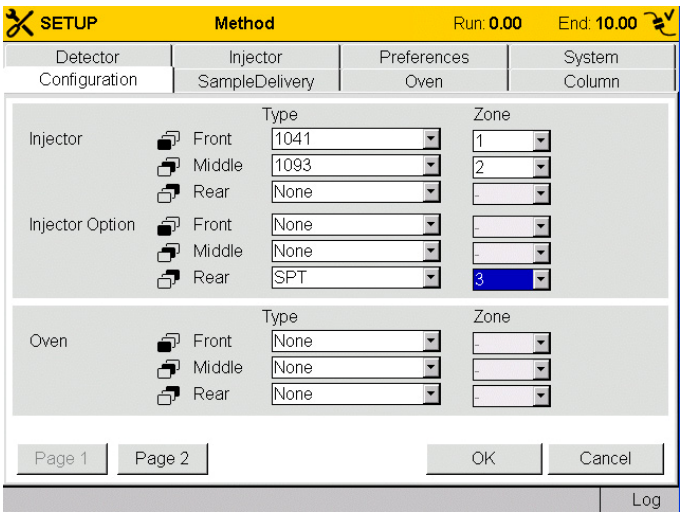
No cryogen is used for trap temperature settings ≥ 50 °C; no heat is applied to the trap at trapping temperatures < 0 °C.

It is advisable to purge adsorbent-containing traps by using an inert gas (N_2 or He). This procedure prevents inadvertent oxidation of the adsorbent by accidental heating of the trap.

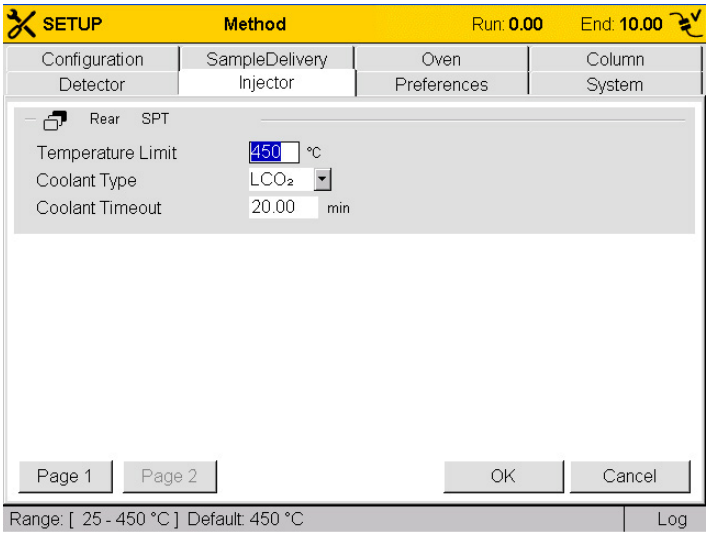
[a] Trapping temperature range is determined by coolant type.
[b] Trapping temperature range is determined by coolant type.

Configuring the SPT

The SPT is typically factory installed and should be already configured when the 450-GC is delivered. To check the SPT configuration press the setup button. The SPT **must** be installed in **zone 3**.



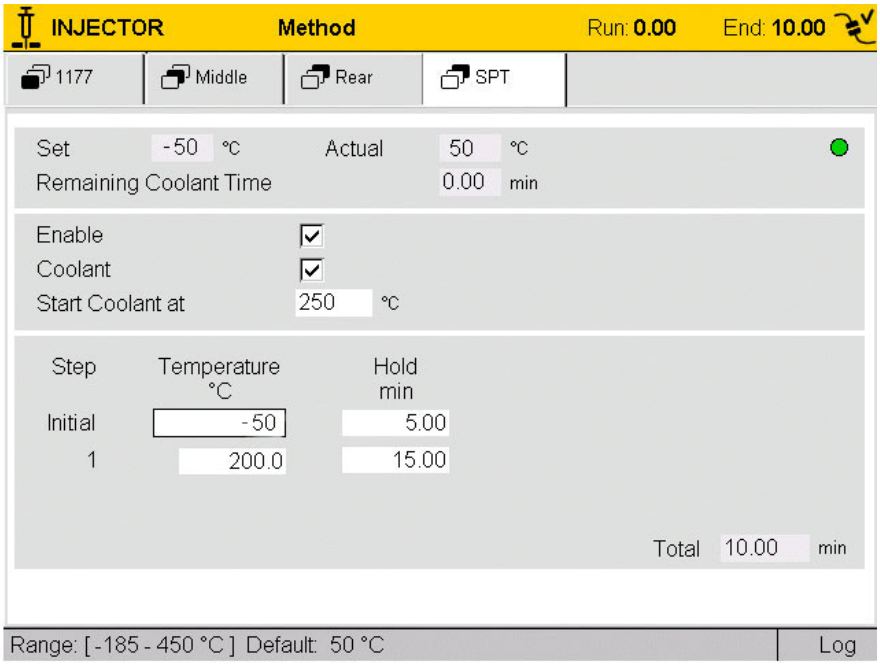
Press on the Injector tab.
Press Page X button to view the SPT Injector setup, verify that the coolant choice of LCO_2 or LN_2 matches the 450-GC hardware.



Building an SPT method

Follow the steps to set the SPT parameters:

1. Press the Injector button and press the SPT tab.



2. Verify that the SPT Enable checkbox is checked.

3. Verify that the Coolant checkbox is checked.

4. The “Start Coolant at” parameter is the temperature at which the coolant valve turns on.

When the cooling starts the “Remaining Coolant Time” will count down. If the cooling reaches the set temperature and the 450-GC does not go into run, the cryo-valve will be turned off it the coolant time-out is reached, and no further coolant will be applied. An event error will then be generated.

5. The SPT temperature program can now be entered, see above for a typical SPT program.

Replacing the Trap Assembly

WARNING: BURN HAZARD

The trap can suddenly become extremely hot. Both the trap and the SPT valve oven can remain hot for an extended period of time, although the SPT cover, cryo enclosure, and SPT valve oven cover might become cool. Always turn power off to the GC and disconnect the SPT trap harness connector from the SPT transformer prior to removing the SPT cover or working on the SPT trap.

Tools required

- 3/16” back-up wrench (supplied in kit)
- 7/16” open-end wrench
- Needle nose pliers
- #2 Phillips screwdriver
- Large flat blade screwdriver (LN_2 Option only)
- 1/4” open-end wrench (2)

CAUTION

Varian’s ultra-clean reducing ferrules (supplied in kit) are required for connecting the 1/16” stainless steel oven/trap lines to the trap ends. These ferrules provide leak-tight seals, while electrically isolating the trap end fittings from the tubing, and have been specially treated to ensure that they are free of contamination.

To maintain the ultra-clean condition of the reducing ferrules, the use of clean cotton gloves or clean forceps is recommended.

SPT System Components

The replacement trap assembly is provided as a single unit. It should not be disassembled. The following description of its basic components is for reference only during installation of a replacement trap assembly.

VTAT traps cannot be used in the SPT.

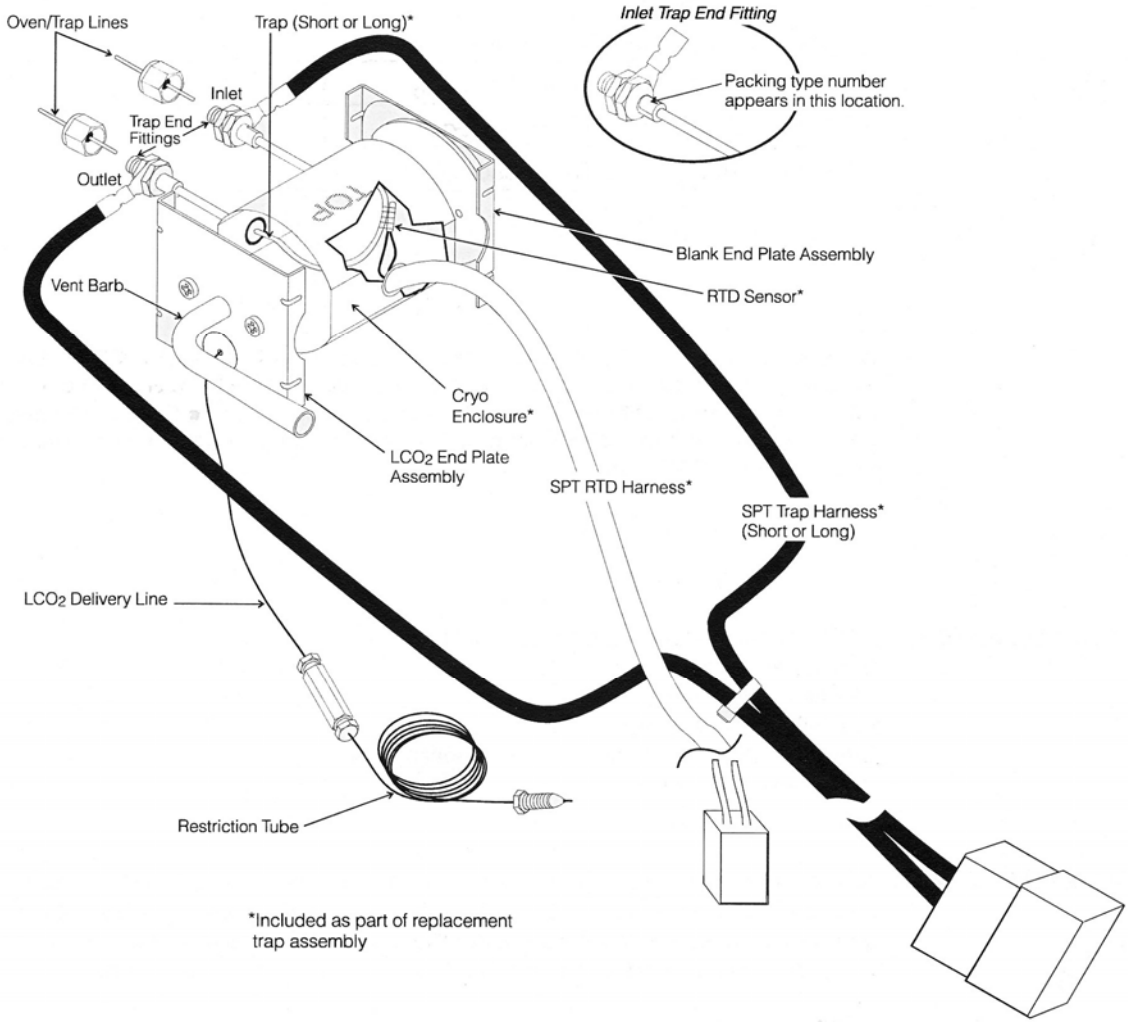
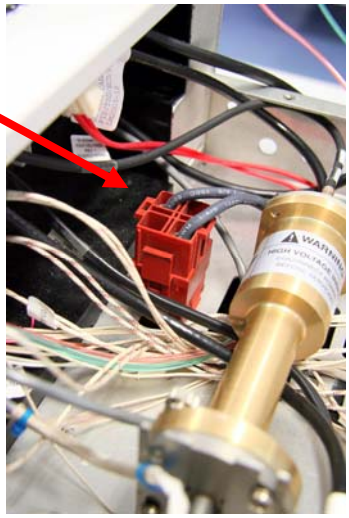


Figure 41: SPT Components (Short Trap Assembly, LCO_2 Option Shown

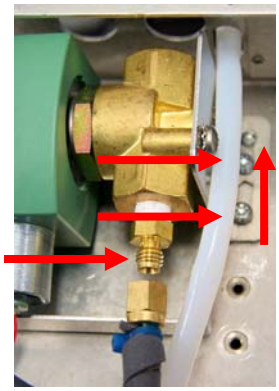
Remove the SPT Cover

Follow these steps to remove the SPT cover and valve oven.

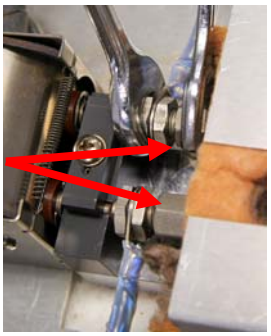
1. Turn the 450-GC power off (using the powerswitch) and unplug power cord.
2. Shut of coolant supply.
3. Remove GC top cover.
4. Unplug SPT trap harness connector from SPT transformer harness (on page 256).
5. Remove SPT cover, by loosen the 2 screws.



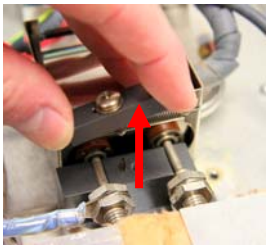
6. Disconnect the **LCO_2 delivery line** (part of **LCO_2 end plate assembly**) from the **union** that connects it to the **restriction tube** or Disconnect **LN_2 delivery line** (part of **LN_2 end plate assembly**) from the **LN_2 solenoid valve** by loosening and disengaging the Swagelok nut from the fitting on the **LN_2 solenoid valve**. The **valve mounting bracket** might need to be loosened slightly so fitting can be moved towards the rear.



7. Disconnect oven/trap lines from **trap end fittings.** Use the 3/16” backup wrench to prevent rotation of the **trap end fittings** (the flats on **trap end fittings** should remain vertical). With the 7/16” open-end wrench, loosen the two **Swagelok nuts** to release any strain on the **reducing ferrules**.



8. Locate the screw holding the trap clamping bar. Notice the Belleville washer under the screw head. Unscrew and remove the bar.



9. Lift the trap/end plate assemblies to disengage trap ends from slots in trap mounting block.

10. Remove the trap/end plate assemblies (if necessary, lift up the SPT valve oven cover).

Detach Trap Assembly

1. Unhook the four **SPT extension springs** from the slots along the edge of the **LCO_2** or **LN_2 end plate**. The springs should remain attached to the **blank end plate assembly**. Use needle nose pliers to gently grip the open hook end of the extension springs. Do not overextend the **springs**. Overextending them might result in their permanent distortion.

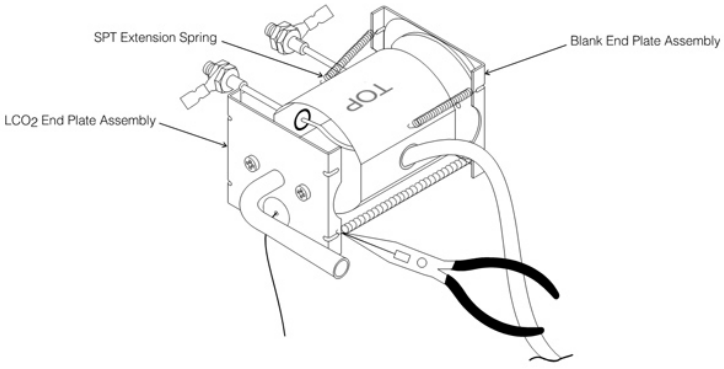


Figure 42: Removing Springs from Trap assembly

Remove Cryo Nozzle Assembly

Follow the next steps to remove Cryo Nozzle Assembly form LN_2 end plate assembly.

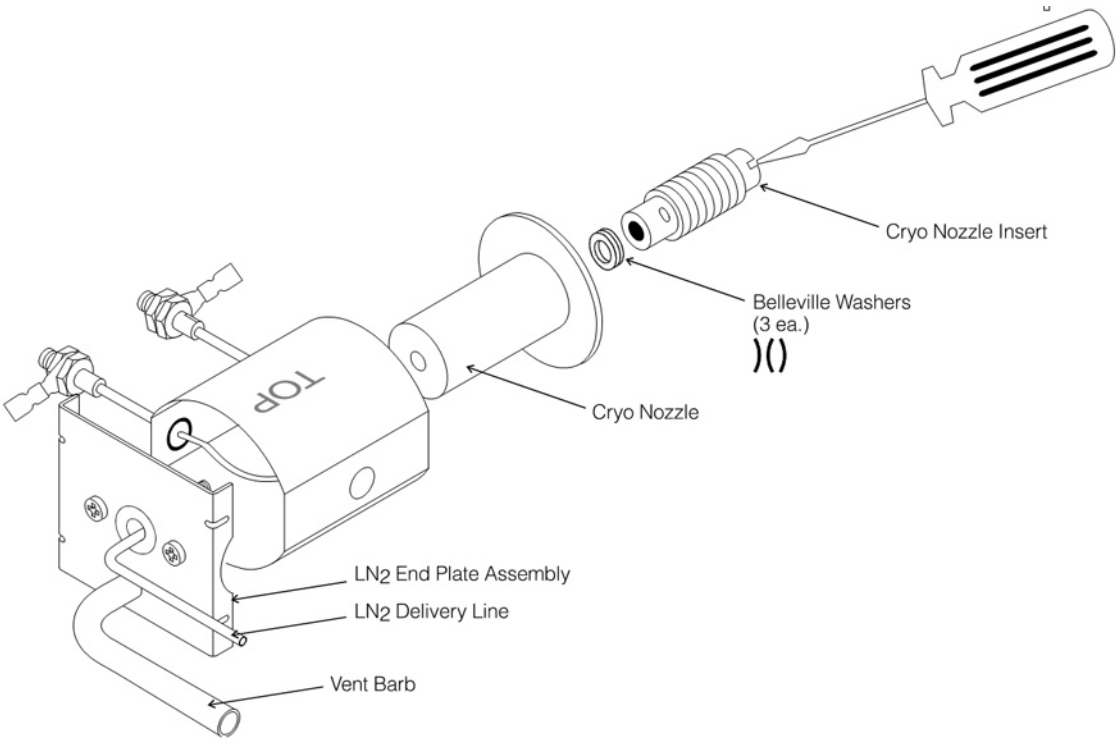


Figure 43: Cryo Nozzle Assembly

1. Unscrew the **cryo nozzle insert** using a large flat bladed screwdriver. Do not remove the **cryo nozzle insert** from within the **cryo nozzle assembly**.

2. Remove entire **cryo nozzle assembly:** hold both **cryo nozzle** and **cryo nozzle insert** together while removing them from within the trap coil. This prevents loss of the three **belleville washers** that are stacked inside the **cryo nozzle assembly**.

CAUTION

If the used trap assembly is still functional, its ends should be capped off to isolate it from atmospheric contamination during storage.

Installing the Trap Assembly

Follow the steps below to prepare the replacement trap:

1. Confirm the part number of the intended replacement **trap assembly**. Check that the size of the trap is correct (the long trap has three coils).

2. Uncap the replacement trap.

3. Hold the **trap assembly** *with the trap ends pointed towards you*, *and the word* "TOP" *on the* **cryo enclosure** *facing upwards*.

   In the following procedures, the terms "right" and "left" refer to this orientation.

4. Preassemble the **LN_2** cryo nozzle components (Figure 43, on page 268):

   a. Using a narrow pointed tool (such as a small screwdriver), align the stack of three **belleville washers** in alternating orientations, placing the first washer with its convex side facing the pointed tool.

   b. Holding the pointed tool pointing up so the washer stack doesn't fall off, lower the **cryo nozzle** over the washer stack so that the washers are inserted fully into the large cavity of the **cryo nozzle.**

   c. Tip the **cryo nozzle** so that its large cavity is facing upwards, then remove the pointed tool so as to not disturb the orientation of the washer stack.

   d. Insert the **cryo nozzle insert** (slotted end facing out) into the cryo **nozzle cavity**.

   e. Keep the **cryo nozzle insert** closely engaged within the **cryo nozzle.**

5. Align the **LN_2** end plate assembly with the right open end of the cryo enclosure and engage the cryo nozzle assembly (Figure 43, on page 268):

   a. Orient the **LN_2 end plate assembly** so that its **delivery tube** is pointing away from you. **Vent barb** and **vent tube** should be at the bottom with **vent tube** exiting to the rear.

   b. The **LN_2 end pilot assembly**, which is attached to the inside of the **LN_2 end plate assembly**, should fit into the right open end of the **cryo enclosure**. Take care not to catch the edge of the **gasket**

against the rim of the **cryo enclosure**; the rim should bear evenly against the **gasket** facing in the area surrounding the **end pilot.**

c. From the left side of the trap coil, engage the **cryo nozzle assembly** through the coil. Screw the **cryo nozzle insert** onto the threaded **stud** of the **LN_2 end pilot assembly** using a large flat blade screwdriver. Take care not to cross-thread the **cryo nozzle insert**. Tighten gently, then back off approximately 1/12th of a turn.

6. To align the **LCO_2** end plate assembly, with the right open end of the cryo enclosure:

   a. Orient the **LCO_2 end plate assembly** so that its **delivery tube** is near the bottom of the right face and pointing away from you. The **Vent barb** and **vent hose** should exiting to the rear.

   b. The **LCO_2 end pilot assembly**, which is attached to the inside of the **CO_2 end plate assembly**, should fit into the right open end of the **cryo enclosure**. Take care not to catch the edge of the **gasket** against the rim of the **cryo enclosure**; the rim should bear evenly against the **gasket** facing.

7. Align the **blank end plate assembly** (see Figure 42, on page 267) with the left open end of the **cryo enclosure**. The four **SPT extension springs** attached to this **assembly** will have to be held out of the way of the **enclosure**. The **blank end pilot** should engage into the left rim of the cryo enclosure. Take care not to catch the edge of the **gasket** against the rim of the enclosure; the rim should bear evenly against the **gasket** facing.

8. Use needle nose pliers to gently grip the open hook end of the **extension springs**, and extend the **springs** to engage their respective slots that should be aligned on the right end of the **cryo enclosure** in the **LCO_2** or **LN_2 end plate assembly**. Do not overextend the **springs**. Overextending them might result in their permanent distortion.

9. If both **end plate assemblies** are properly engaged into the **cryo enclosure**, they should be parallel to each other and perpendicular to the sides of the cryo enclosure. The **SPT extension springs** should firmly hold the **end plate assemblies** against the rims of the **cryo enclosure**. The **SPT extension springs** should not touch the trap ends.

10. The oven/trap lines that connect to the **trap end fittings** should be preformed for proper alignment with the **trap end fittings**. The **nut** and the **reducing ferrules** should be preplaced onto the line ends so that when the trap is installed, there is minimal bending of the lines.

To maintain the ultra-clean condition of the reducing ferrules during installation, the use of clean cotton gloves or clean forceps is recommended.

11. Confirm that the **trap assembly** with the trap ends pointing towards the front of the GC, with the word “TOP” on the **cryo enclosure** facing upwards.

12. Engage the oven/trap lines into trap ends.

13. Align both trap ends with the slots in the **trap mounting block**. Lower trap ends to engage into the slots.

14. Place the **trap clamping bar** over the trap ends.

15. Make sure the belleville washer on the trap clamping bar screw has its concave side facing the trap clamping bar. Tighten **trap clamping bar screw** to anchor the **trap assembly** into the **SPT valve oven**. Do not overtighten.

16. Reconnect the valve oven/trap lines to **trap end fittings**. The packing type number is stamped on the inlet **trap end fitting** (see Figure 41 on page 265). Carefully engage each **nut** onto its respective **fitting** to prevent cross-threading. Using the faces of the hexagonal nut as a guide, tighten each nut fingertight, then tighten an additional:
    a. Using new ferrules, 4/6 - 5/6 of a turn.
    b. Reusing ferrules, 2/6 of a turn.

17. Do not over tighten. Use 3/16” backup wrench to prevent rotation of the **trap end fittings** (the flats on **fittings** should remain vertical).

CAUTION

To avoid the possibility shorting the SPT electrical circuit, do not insert the valve oven/trap lines more than 8 mm into the trap ends. Inserting the lines too far into the trap ends will cause them to come in contact with the trap.

CAUTION

Do not plumb the valve in such a way that metal tubing shorts across trap tube ends.

18. Connect the **LCO_2** delivery line:

    a. Connect the **LCO_2 delivery line** (part of **LCO_2 end plate assembly**) to the union that connects to the **restriction tube**.

    b. Check that **vent barb** and **vent hose** are exiting to the rear. Feed the **vent hose** through the back panel of the GC.

19. Connect the **LN_2 delivery line** (part of **LN_2 end plate assembly**) to the **LN_2 solenoid valve** by engaging the Swagelok nut to the connector on the valve (see on page 266). Do not over tighten Swagelok fitting. The **Valve mounting bracket** might need to be anchored to the GC. Do not induce strain onto the **delivery line**: carefully align the **LN_2 solenoid valve** and its bracket during tightening of the **valve mounting bracket.**

20. Check that the **drip pan** is located under the **LN_2 solenoid valve**. The **drip pan** should not excessively block vent holes in the column oven top.

21. Tighten down the SPT valve oven cover.

22. Install the SPT cover.

23. Connect the SPT trap harness connector to the SPT transformer harness.

24. Replace GC top cover.

25. Turn on coolant supply.

26. Power up GC.

Trap Conditioning

With carrier or auxiliary gas passing through the trap, condition the trap according to the following table:

Packing Type	*Mesh Size*	*Conditioning Procedure*
Silanized Glass Bead	60-80	1 to 16 hours @ 250 to 400 °C
Tenax/GR Charcoal	60-80	4 to 16 hours @ 250 °C
HayeSep D	60-80	4 to 16 hours @ 250 °C
Carbopack B, Carbopack C, Carboxen 1000, Carboxen 10001	60-80	4 to 16 hours @ 270 °C
5% OV-101 on Chromosorb G/HP Haysep D	60-80	4 to 16 hours @ 270 °C
Tenax Gr, Carbopack B, Carbosieve S-111	60-80	4 hours @ 225 °C
Tenax TA,	60-80	4 hours @ 225 °C
Tenax TA, Charcoal	60-80	4 hours @ 200 °C
Carbopack C, Carbosieve S-111	60-80	4 hours @ 200 °C
Carbopack C, Carbopack B	60-80	4 hours @ 270 °C
Carbopack B, Carbosieve S-111	60-80	4 hours @ 200 °C
Tenax GR	60-80	4 hours @ 200 °C

Table 49: SPT Trap conditioning

Replacement SPT traps

The table below is a partial list of available trap assemblies. Consult your local sales representative or Parts and Supplies Marketing for additional versions.

Trap Description	***Partnumber***
Short Traps	
Short Trap Assembly Kit (8 cm), silanized glass beads	03-925713-92
Short Trap Assembly Kit (8 cm), blank	03-925713-91
Blank Trap	03-925713-01
Packed, Glass Beads	03-925713-02
Long Traps	
Long Trap Assembly Kit (30 cm), blank	03-925714-91
Blank Trap	03-925714-01
Tenax GR/Charcoal	03-925714-02
Tenax GR	03-925714-03
Carbopack B, Carbosieve S-III	03-925714-04
5% OV-101 on Chromosorb G/HP, HayeSep D	03-925714-05
Carbopack C, Carbopack B, Carboxen 1000, Carboxen 1001	03-925714-06
Carbopack C, Carbosieve S-III	03-925714-07
HayeSep D	03-925714-08
Tenax TA, Charcoal	03-925714-09
Tenax TA	03-925714-10
Tenax GR, Carbopack B, Carbosieve S-III	03-925714-11
Kit, Blank Trap	03-925714-91
Kit, Tenax GR/Charcoal	03-925714-92
Kit, Tenax GR	03-925714-93
Kit, Carbopack B, Carbosieve S-III	03-925714-94
Kit, 5% OV-101 on Chromosorb G/HP, HayeSep D	03-925714-95
Kit, Carbopack C, Carbopack B, Carboxen 1000, Carboxen 1001	03-925714-96
Kit, Carbopack C, Carbosieve S-III	03-925714-97
Kit, HayeSep D	03-925714-98
Kit, Tenax TA, Charcoal	03-925714-99
Kit, Tenax TA	03-925714-80
Kit, Tenax GR, Carbopack B, Carbosieve S-III	03-925714-81

Table 50: SPT replacement traps

SPT replacement parts

Part Description	Partnumber
Cryo Valve: LCO_2 LN_2	03-925555-01 03-925557-01
Ferrule for Restriction Tube (LCO_2)	28-694502-00
Nuts for Restriction Tube (LCO_2)	28-694501-00
Insulation Tubing (LN_2)	24-000467-00
Outlet Filter (LCO_2)	27-590826-00
Reducing Ferrule (Ultra-clean, for Trap Assembly)	03-918850-00
Restriction Tube, 22” Long (LCO_2)	03-918853-01
SPT PCB	03-918758-00
SPT Transformer	03-918788-00
Transfer Line from LN_2 Supply to GC (1/4” Copper, 10’ Length)	37-000146-01
Trap Clamping Bar	03-918765-00
Trap Mounting Block	03-918764-00
Union (LCO_2)	28-211457-00
Transfer Line from LCO_2 supply to GC (1/16” stainless steel, 10’)	03-918852-00
Ferrules for stainless steel Transfer Line (Front)	28-693996-00
Ferrules for stainless steel Transfer Line (Back)	28-693997-00
Nuts for stainless steel Transfer Line	28-693998-00
Ferrules for stainless steel Transfer Line (1/8”) (Front)	28-694027-00
Ferrules for stainless steel Transfer Line (1/8”) (Back)	28-694028-00
Nuts for stainless steel Transfer Line (1/8”)	28-694029-00
Ferrules, Vespel, Reducing 1/8” To 1/16”	03-918850-00
Installation Kit, LCO_2	03-918801-01
Installation Kit, LN_2	03-918801-02

Table 51: SPT Replacement Parts

Troubleshooting

This section provides troubleshooting information to aid the operator in locating and correcting apparent problems when working with an SPT system. The following table is geared specifically to SPT troubleshooting. If you have questions about any of the corrective actions described below, contact your Varian Customer Support Representative.

Symptom	***Possible Cause***	***Remedy***
SPT overheating.	Faulty SPT RTD sensor. SPT RTD harness not properly connected to SPT electronic board.	Replace trap assembly. Check and reconnect harness.
SPT heating too slowly.	*Short in electrical system:* Oven/trap line making contact with trap. Insulating surface (gray) of the SPT trap mounting block damaged. Bushings not isolating trap from the cryo enclosure. Cryo valve stuck open.	 Adjust oven/trap line and trap end fitting. Replace mounting block. Replace trap assembly. Replace cryo valve.
SPT not heating.	SPT transformer circuit breaker tripped. SPT trap harness not properly connected to the SPT transformer. SPT RTD harness open or shorted. SPT trapping temperature set to <0° C.	Correct problem; reset. Reconnect harness. Replace trap assembly. Set temperature to equal or greater than 0°C.

Symptom	***Possible Cause***	***Remedy***
No coolant flow.	SPT performing desorption temperature program. Coolant supply exhausted or low. Coolant restriction tube plugged (LCO_2). Coolant supply not connected. Coolant has timed out (status light blinking). Coolant is not enabled in GC Configure table. Cryo valve is not connected to Temperature Control electronic board. Cryo valve stuck closed.	Perform trapping temperature program. Replace coolant supply. Clean or replace restriction tube. Replace in-line filter. Connect and turn on coolant supply. Press [RESET]; Enter new value for Coolant Timeout, if desired. Enable coolant. Connect cryo valve. Replace cryo valve.
SPT overcooling.	Cryo valve stuck open.	Replace cryo valve.
SPT undercooling or cooling too slowly.	Supply cylinder too warm or to cold. Coolant supply has been depleted of liquid phase CO_2.	Relocate coolant supply to a location within recommended temperature limits. Make sure that GC and transfer line are cooler than cryo supply cylinder. Replace supply with a full dewar or cylinder.
Trapping temperature is oscillating out of ready.	Supply too warm (≥27°C) GC and transfer line are too warm relative to the supply.	Relocate coolant supply to a location within recommended temperature limits. Make sure that GC and transfer line are cooler than cryo supply cylinder. Provide a cooler environment for the GC (should be cooler than cryo supply).

Symptom	***Possible Cause***	***Remedy***
	Transfer line is too long.	Shorten transfer line. Insulate transfer line.

DETECTORS

INTRODUCTION

The 450-GC can accommodate up to three installed detectors and have all three running simultaneously.
The standard detectors available on the 450-GC are:

- The Flame Ionization Detector (FID), on page 283.
- The Pulsed Discharge Helium Ionization Detector (PDHID), on page 295.
- Thermionic Specific Detector (TSD), on page 305.
- Thermal Conductivity Detector (TCD), on page 319.
- Electron Capture Detector (ECD), on page 329.
- Pulsed Flame Photometric Detector (PFPD), on page 343.

Except for the TCD and PFPD any combination of three detectors can be installed.
In case of the PFPD, only two may be installed but any one additional detector may also be installed including a TCD.
In the case of the standard TCD, only two can be installed and then no additional detectors can be installed.
However, a dual TCD option is available as a custom solution which allows installation of an additional ionization detector.
The dual TCD consists of 2 detector cells in one housing but using only one heating zone.

Detectors are mounted on the top of the 450-GC, above the left side of the column oven.
The position of the detector, however, is determined by the location of the detector's electrometer in the electronics cabinet. Detector electrometers are installed on the left side of the GC accessed by removing the left side panel.
All cables connecting the electrometers to the detectors are accessible by removing the 2 covers on the top left of the 450-GC, i.e. the detector top cover and the cover over the keyboard/display.

For connecting a capillary column to a detector, see Connect Capillary Column to Detector on page 24.

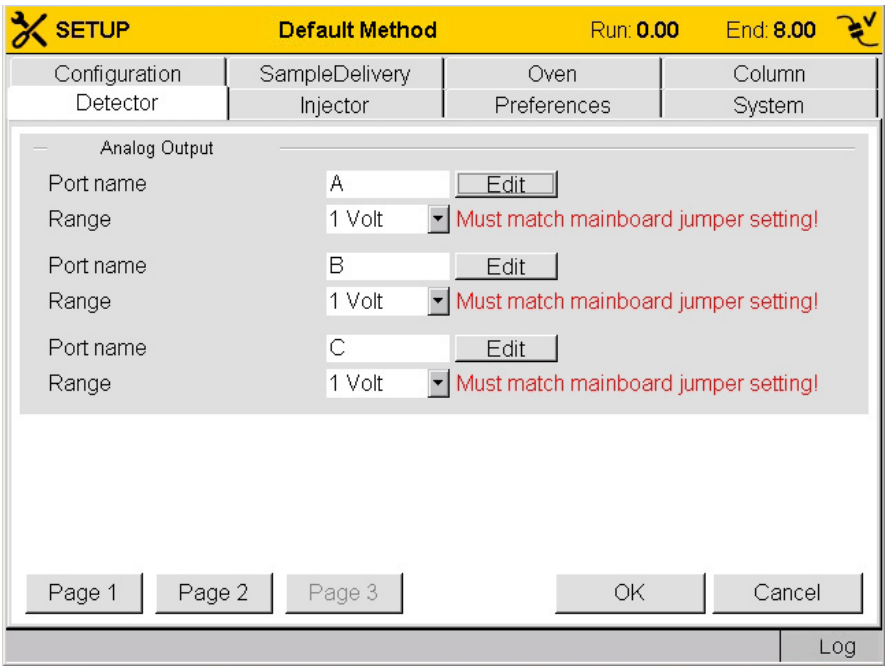
ANALOG OUTPUT

The 450-GC can be equipped with an option board.
The analog output signals are located at connector J401, on page 36 on the top of the GC, accessed by removing the top left cover.
This connector contains all 3 analog channels plus status signals for “Ready-In” to the GC and “Start Out” from the GC.

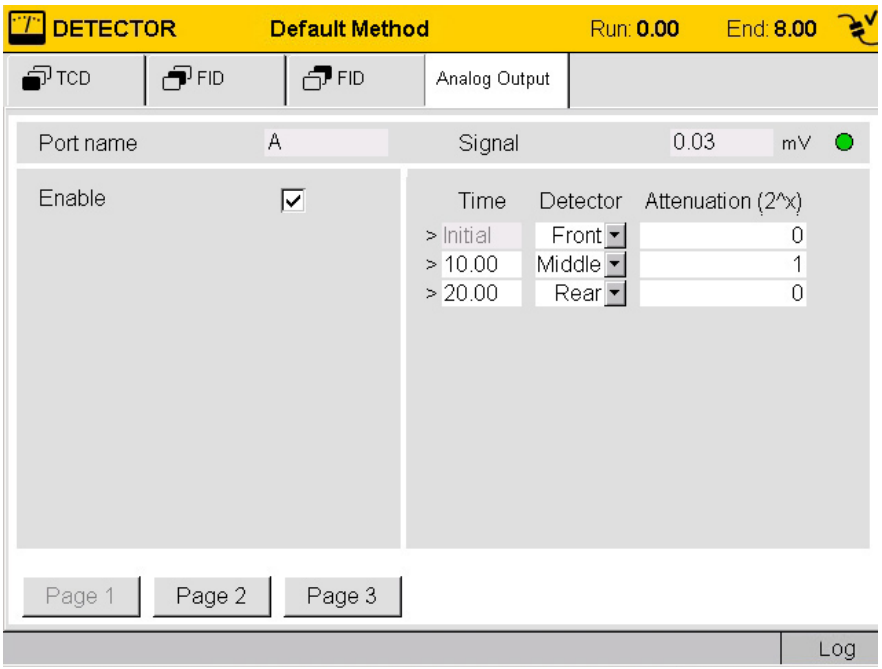
In the SETUP page by the detector tab on the second page (Page 2 or page 3) the Detector Analog Output screen will be visible, only if the Analog option board is present.

The Analog Output Port name can be changed by the button “Edit”.

If the Range setting must be changed, make sure that the Range setting matches the mainboard jumper settings!



The analog output ports are programmed in the 450-GC method by pressing the DETECTOR key on the keyboard, select Analog Output.
This brings up a menu screen from which you choose one of the three ports to program; Page 1, Page 2 or Page 3. The following screen appears when Page 1 is chosen.



The Analog Output Port name is in this example is A, but can be changed in the Setup screen. The individual analog ports can be assigned to any installed detector on the 450-GC. The ports are identified as A, B and C with the default assignment being front, middle and rear detector respectively. Default Analog output voltage is 0-1 Volt (0-10 Volt is possible by selectable jumpers on the mainboard (contact your local Varian office).

The right-side of the screen is used to time program the signal source of the analog output. By default analog output A is assigned to the front detector. In the example above, the analog port is programmed to transmit the Front detector signal for the first 10 minutes of the run and then switch to the Middle detector after 20 minutes the switch will be made to the rear detector for the remainder of the run. This mode of operation is known as detector switching.

Note that the attenuation should normally be set to 0. However for optimum resolution (less electronic noise) the best results are achieved by increasing the attenuation if small peaks are detected. For overloaded peaks decrease attenuation.

The 450-GC has a number of analog cable options on page 94, depending on the device to which the cable is connected. All cables have a 15 pin D-shell connector on one end to attach to J401 on the 450-GC and have the appropriate connectors on the other end of the cable for the devices to which they are being attached.

FLAME IONIZATION DETECTOR (FID)

The flame ionization detector (FID) is the most sensitive gas chromatographic detector for hydrocarbons such as butane or hexane. With a linear range for 6 or 7 orders of magnitude (10^6 to 10^7) and limits of detection in the low picogram or femtogram range, the FID is the gas chromatographic detector for volatile hydrocarbons and many carbon containing compounds.

The FID detector employs hydrogen as the combustion gas which is mixed with Air and the column eluent (helium, nitrogen or other appropriate gas) and burn at a small jet situated inside a cylindrical electrode. A potential of a few hundred volts is applied between the jet and the electrode and when a carbon containing solute is burn in the jet, the electron/ion pairs that are formed are collected at the jet and cylindrical electrode.

The following instructions refer to the operation of a 450-GC Flame Ionization Detector (FID). The FID is installed on a detector base, directly above the column oven. The FID electrometer is installed in the electronics cabinet on the left of the instrument. The electronics of the FID are controlled from the 450-GC Display/Keyboard, the gas flows are set and controlled either from the 450-GC Display/Keyboard (if detector EFC is installed) or at the pneumatics panel.

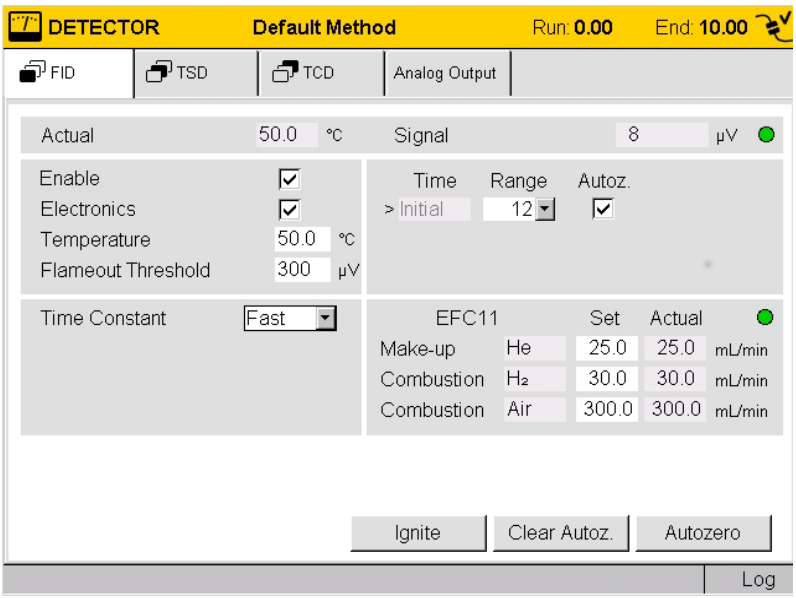
Initial Set-Up

To set-up the FID, proceed as follows. Note that when the 450-GC is first powered up all heated zones with the exception of the column oven are powered off and all detector electronics are turned off.

1. Refer to the Pre-Installation Instructions for additional information on gas supply requirements.

2. Press the DETECTOR key on the 450-GC Display/Keyboard to display the DETECTOR screen. The FID should be listed in position Front (), Middle () or Rear (). If your GC is equipped with detector EFC verify the makeup gas you will be using with your FID.

3. Install a chromatographic column in the GC, on page 13. If the analytical column is not pre-conditioned, use a no-hole ferrule in the detector column nut while conditioning the column and leave the detector end of the column loose in the oven. If the analytical column is pre-conditioned, follow the usual procedure for column installation.



4. If detector EFC is present, set the following flow rates in the adjustments section of the FID method:

   - Make-up flow to 25 mL/min.
   - The hydrogen flow to 30 mL/min.
   - Air flow to 300 mL/min.

   Note: For optimum performance the combined column + make-up flow should be 30 mL/min, so some adjustment of the make-up flow may be necessary.

5. If manual pneumatics is installed, attach a bubble or digital flowmeter (AL5700) to the top of the FID tower using the adapters supplied in the FID accessory kit (Note that the most reliable measure of flow is directly from the flame tip. This prevents wrong-adjustment due to leaks in the overall tower assembly). Check the following flow rates and set them if necessary: combined carrier gas + make-up flow to 30 mL/min, the hydrogen flow to 30 mL/min and the air flow to 300 mL/min.

6. Press the DETECTOR key on the 450-GC Display/Keyboard, select the FID by its location and turn on the FID oven power. Set the oven temperature to 300 °C. Verify that the FID electronics is turned OFF.

Operation

1. Check the detector temperature status on the DETECTOR/FID screen. Check the FID range setting and set to range 12, if necessary.

Generally, to prevent water condensation in the detector assembly, the detector should be operated at a temperature above the column temperature and not below 150 °C. If the detector is operated at a lower temperature, condensation can lead to excessive noise.

2. After the FID oven has reached its setpoint temperature, turn on the FID electronics. Note that the FID will ignite automatically when auto-ignite conditions are fulfilled (for detailed information about auto-ignite conditions see here, on page 287). The 450-GC monitors the FID background current continuously and will attempt to ignite the flame (only if in Setup/Detector the checkbox on page 75 “check for flame out” is checked) if the current drops below a specified threshold. Up to three attempts to ignite the flame will be made before the 450-GC reports a flame-out fault. If a flame-out fault is reported, correct the cause of the flame-out and then clear the fault in Instruments Log.

   The FID will automatically ignite after the fault is cleared.

WARNING:
EYE HAZARD

DO NOT look directly into the detector tower when attempting to ignite the flame.

You can verify that the flame is lit by monitoring the FID signal in the status region of the FID display.
Uncheck the box “AutoZ.” and press “Clear AutoZ.”
Initially the signal will read a very high value but should then stabilize. Typically the signal will be > 50 mV at range 12 when the flame is first lit but should drop to < 10 mV within 30 minutes.

WARNING:
EXPLOSION HAZARD

To avoid a possible fire or explosion, always turn off the flow of hydrogen when the column is removed or when the detector is not being used. This prevents the accumulation of hydrogen.

3. Set the Time Constant to “Fast”. The default setting for the time constant is “Fast”.

   Fast time constant:
   - Used with capillary columns.
   - Reduce high frequency noise.
   - Used for narrow peaks.

   Slow Time Constant:
   - Used with packed columns.
   - Used for broad peaks.

4. The “Ignite” button applies power to the FID igniter filament for 5 seconds.

5. The “Autozero” button applies an immediate zeroing of the detector signal.

FID auto-Ignite flow diagram

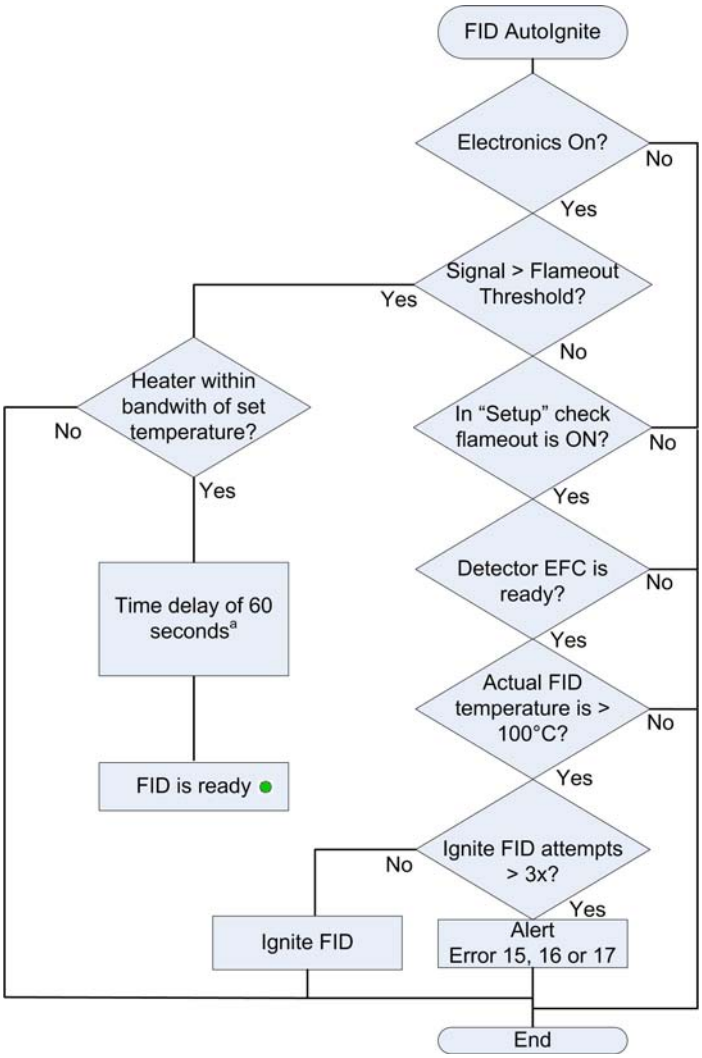


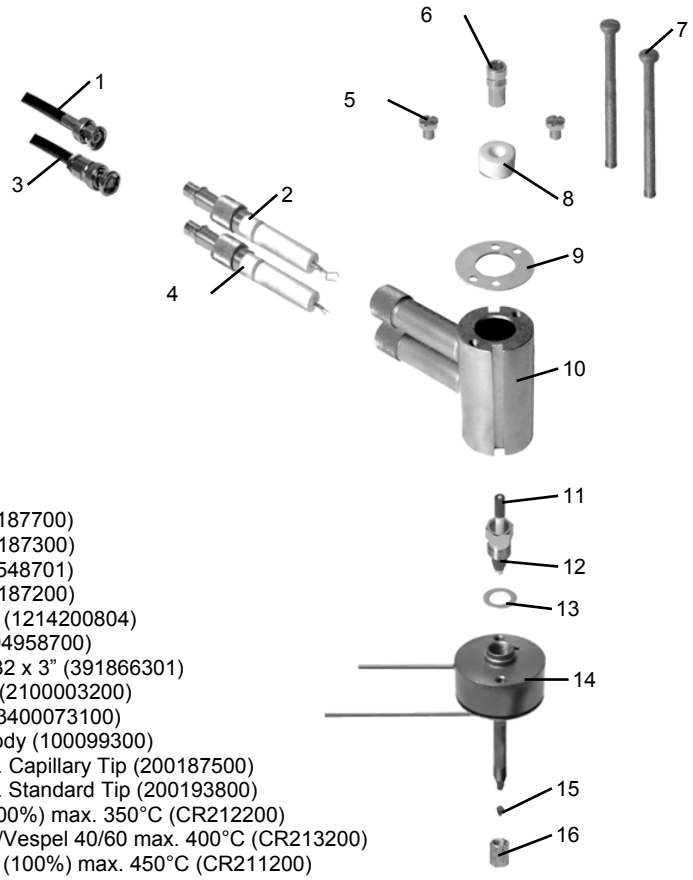
Figure 44: FID auto-Ignite flow diagram

[a] In an automated sequence the 60 seconds delay helps to recover from FID flame faults and avoids having the fault again in the next run. It also prevents a workstation starting a run while FID ignition is still going on.

Installation/Disassembly

At times, it is necessary to remove the detector components to inspect, to clean, to replace parts, or to install another detector. Follow the disassemble/reassemble instructions on the next pages for both the FID and the detector oven.

Exploded View



1 Signal Cable (200187700)
2 Signal Probe (200187300)
3 Ignitor Cable (392548701)
4 Ignitor Probe (200187200)
5 Screws, 8-32 x ¼" (1214200804)
6 Collector Tube (394958700)
7 Tower Screws, 8-32 x 3" (391866301)
8 Ceramic Insulator (2100003200)
9 Tower Top Plate (3400073100)
10 Detector Tower Body (100099300)
11 Flame Tip, 0.01 in. Capillary Tip (200187500)
11 Flame Tip, 0.02 in. Standard Tip (200193800)
12 Ferrule: Vespel (100%) max. 350°C (CR212200)
Graphite/Vespel 40/60 max. 400°C (CR213200)
Graphite (100%) max. 450°C (CR211200)
13 Aluminum Seal Washer (1500334701)
14 Detector Base (392547501)
15 Column Ferrule
16 Column Nut (394955100)
--- Flow Tube (200187600)
--- Large Stopper (394958600)

Figure 45: FID Exploded View

Disassemble the FID

To disassemble the FID, proceed as follows:

1. Set the FID to 50 °C and wait for it to cool before disassembly. Turn the detector electronics and detector oven OFF in the active method. Remove the GC Top-Covers.

2. Turn off the main GC gas supplies to the detector at the pneumatics panel. These are the make-up, hydrogen and air supplies.

3. Disconnect the signal and igniter cables from their probes on the detector tower.

4. Remove the signal and igniter probes from the detector tower. *DO NOT* rotate the probes as you disconnect them from the electrical contacts in the tower. Place the probes on a clean surface such as a lint-free laboratory tissue.

5. Remove the 2 tower mounting screws from the top of detector tower.

6. Remove the tower assembly from the detector oven, lifting straight up until clear of the flame tip. Remove the collector tube and insulator from the detector tower. Avoid contamination of the ceramic insulator and probes. If the detector is not completely cool, use a metal tool (such as tweezers or a hooked wire) to remove parts from the tower assembly. Place parts on a clean Kimwipe®. Never place them on a counter or painted surface.

7. Remove the flame-tip assembly from the detector base. Take care not to break the ceramic flame tip tube or the Vespel®/graphite or graphite ferrule.

8. Remove and discard the aluminum seal washer from the detector base. Always use a new aluminum seal washer each time you reassemble the detector. Note that the aluminum seal washer may remain in the detector tower when the tower is removed from the base.

Reassemble the FID

Refer to Figure 45 on page 288 to reassemble the FID detector. To reassemble the FID, proceed as follows:

1. Re-assembly is the reverse of removal.

2. Install the flame tip in the detector base. The FID and TSD flame tips are identical.
   - If you are installing the flame tip with a new Vespel/graphite ferrule, tighten the assembly finger-tight plus an extra 1/6-turn.
   - If you are installing the flame tip assembly with a used graphite ferrule, tighten about 1/3-turn past finger tight.

3. The Vespel/graphite ferrule supplied with the GC has a maximum temperature limit of 400 °C. If it is necessary to operate the detector above 400 °C, you may need to replace the Vespel/graphite ferrule with a graphite ferrule (450 °C).

4. Install a new aluminum seal washer onto the shoulder of the detector base. To ensure a reliable tower seal, use a NEW aluminum washer each time you install the detector.

5. Place the detector tower on the detector base and secure it with the 2 tower mounting screws. Alternately tighten these screws a ½-turn as the tower tightens into place.

CAUTION

Handle the ceramic insulator and probes with tweezers to avoid contamination.

6. Carefully insert the igniter probe into the lower arm of the detector tower. Align the probe key with the tower arm slot. Check the orientation of the igniter elements by looking down through the top of the detector tower. The spring clip should slip around the flame tip and make good contact (see Figure 46 on page 291). The igniter coil must not touch the flame tip assembly nor be positioned directly above it. Tighten the knurled nut to secure the probe.

7. Make sure the notch in the detector tower arm does not cut the O-ring seal.

8. Insert the insulator into the detector tower, then insert the collector tube into the tower. The collector tube must not touch the igniter coil.

9. Insert the signal probe into the upper arm of the detector tower. The probe clip should fit around the tapered section on the collector tube tightly enough to exert a downward force (see Figure 47). Secure the signal probe by tightening the knurled nut. Make sure the notch in the detector tower arm does not cut the O-ring seal.

10. Connect the igniter cable to the igniter probe and the signal cable to the signal probe.

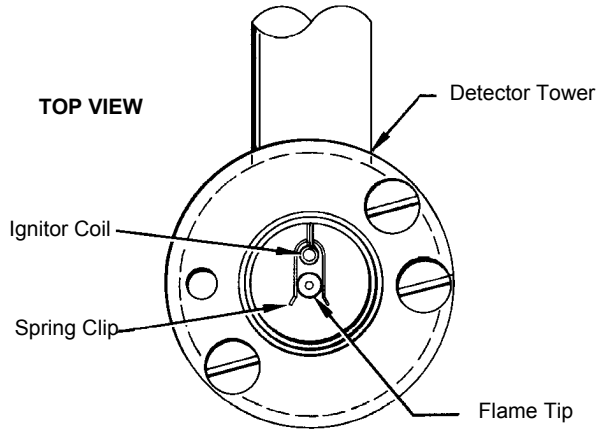


Figure 46: Orientation of Igniter Probe Elements in FID Tower

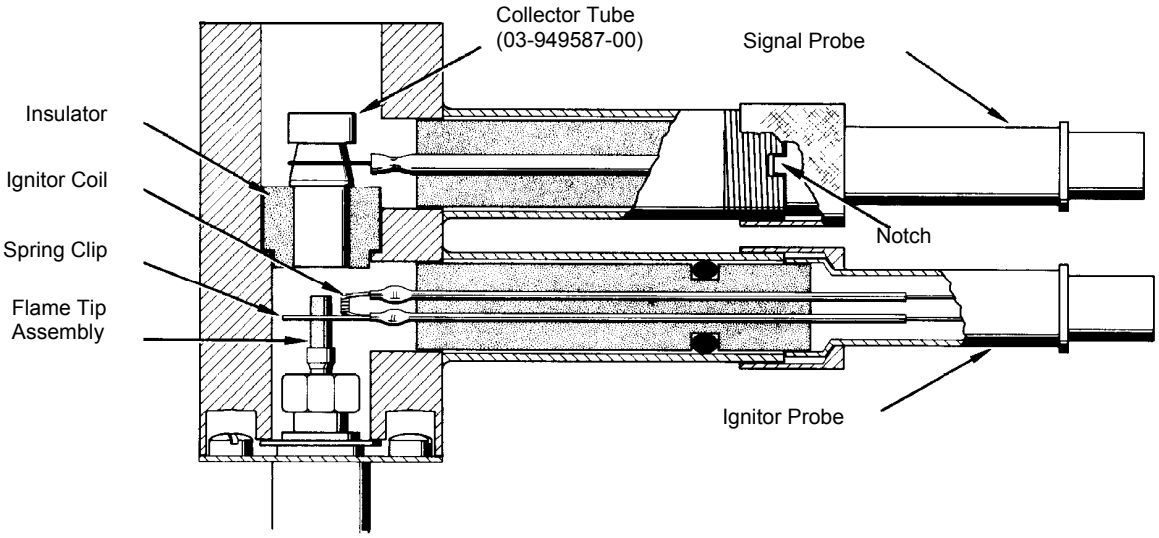


Figure 47: FID Cross-sectional View

MAINTENANCE

When the performance of the FID has degraded you may want to replace the flame tip ferrule, or clean the deposits from the internal parts, including the flame tip.

Clean the Flame Tip and Internal Parts

If the FID noise increases with frequent spikes, the internal parts may be contaminated and need to be cleaned.
To clean the flame tip and the internal parts of the FID, proceed as follows:

Always handle detector components with tweezers to avoid contamination.

WARNING: EYE HAZARD

WARNING: FIRE HAZARD

Use proper eye and skin protection. Methanol and acetone are toxic and flammable chemicals. Exercise appropriate care when you use and dispose of these substances.

1. Remove the detector and internal parts (see disassemble the FID, on page 289).
2. Scrape the deposits from the bore of the collector tube, the insulator and the metal part of the flame tip with emery cloth (e.g., SiO_2).
3. If the flame tip is plugged, insert a wire through the flame tip orifice to clear it.
4. If you have access to an ultrasonic cleaner, clean the collector, insulator and flame tip with distilled water.
5. Rinse the cleaned components with methanol or acetone and air dry.
6. Wipe the detector tower with acetone.
7. Clean the probe arms with methanol and air dry or dry in an air oven (maximum drying temperature of 150 °C).
8. Reassemble the FID, see Reassemble the FID, on page 290.

Replace the Flame-Tip Ferrule

After a period of prolonged use, the flame-tip ferrule may deteriorate and crack. As a consequence, a leak may develop around the base of the flame tip assembly that causes unstable baseline noise and reduced sensitivity. In this situation, or when the detector is to be operated at temperatures above 400 °C, you must replace the ferrule. Be sure to select the proper ferrule for the application. The Vespel/graphite ferrule has a maximum temperature limit of 400 °C see Figure 45on page 288.
Varian recommends to NOT using other ferrules then those listed. Due to small dimensional variations common in these ferrules leaks on higher temperatures are possible.

FID Test Chromatogram

For an FID test chromatogram see section Detector Test Chromatograms, on page 420.

PULSED DISCHARGE HELIUM IONIZATION DETECTOR (PDHID)

The PDHID is a non-radioactive pulsed discharge ionization detector (PDHID) which is optimized for the Varian 450-GC. A schematic representation of the detector is shown in Figure 48.

The detector utilizes a stable, low power, pulsed DC discharge in helium as the ionization source. Elutants from the column, flowing counter to the flow of helium from the discharge zone, are ionized by photons from the helium discharge above. Resulting electrons are focused toward the collector electrode by the 2 bias electrodes.

The principal mode of ionization is photoionization by radiation arising from the transition of diatomic helium He_2 ($A^1\Sigma^+_u$) to the dissociative $2He(1S^1)$ground state. This is the well-known Hopfield emission. The photon energy from the He_2 continuum is in the range of 13.5 to 17.7 eV.

The detector is essentially non-destructive (0.01 - 0.1% ionization) and highly sensitive. The response to organic compounds is linear over five orders of magnitude with minimum detectable quantities (MDQs) in the low picogram range. The response to fixed gases is positive (the standing current increases), with MDQs in the low ppb range.

Detector response is universal except for neon, which has an ionization potential of 21.56 eV. Since this potential is close to the energy of the He* metastable (19.8 eV) but greater than the photon energy from the He_2 continuum, neon exhibits a low ionization efficiency and low detector response. When a dopant is added to the discharge gas, the detector also functions as a selective photoionization detector. (Suitable dopants include Ar for organic compounds, Kr for unsaturated compounds, or Xe for polynuclear aromatics).

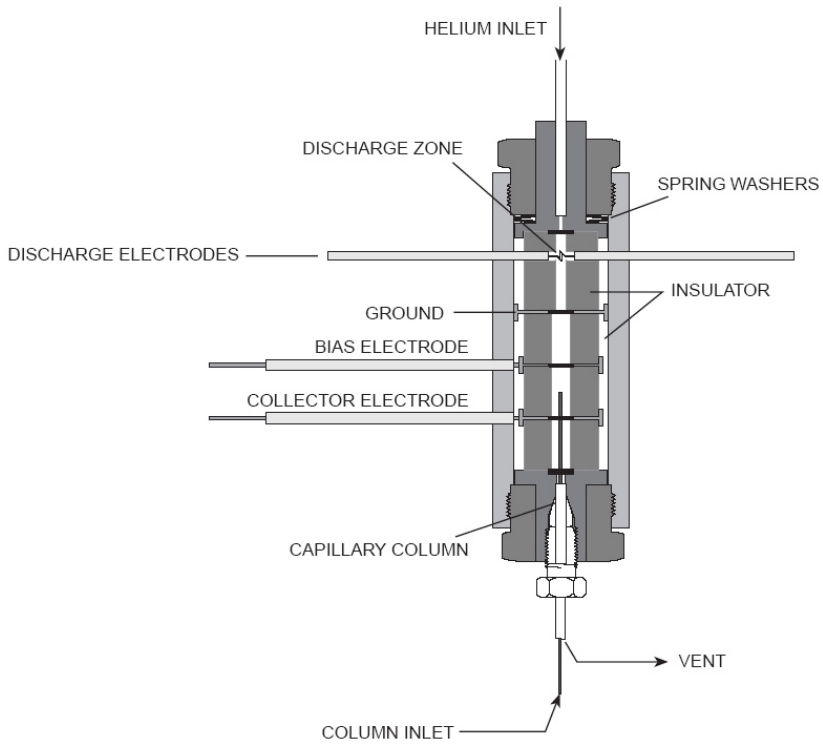


Figure 48: Schematic of the PDHID Detector

HELIUM IONIZATION (PDHID)

The PDHID is essentially non-destructive (0.01 - 0.1% ionization) and highly sensitive. The response to organic compounds is linear over five orders of magnitude with minimum detectable quantities (MDQs) in the low or sub picogram range. The response to fixed gases is positive (the standing current increases), with MDQs in the low ppb range.
The PDHID response is universal except for neon, which has ionization potential of 21.56 eV. Since this potential is close to the energy of the He* metastable (19.8 eV) but greater than the photon energy from the He_2 continuum, neon exhibits a low ionization efficiency and low detector response.

WARNING

During normal operation, the detector produces ultraviolet energy (UVA, UVB), some of which may be emitted. Do not watch the arc without eye protection!

Cable overview

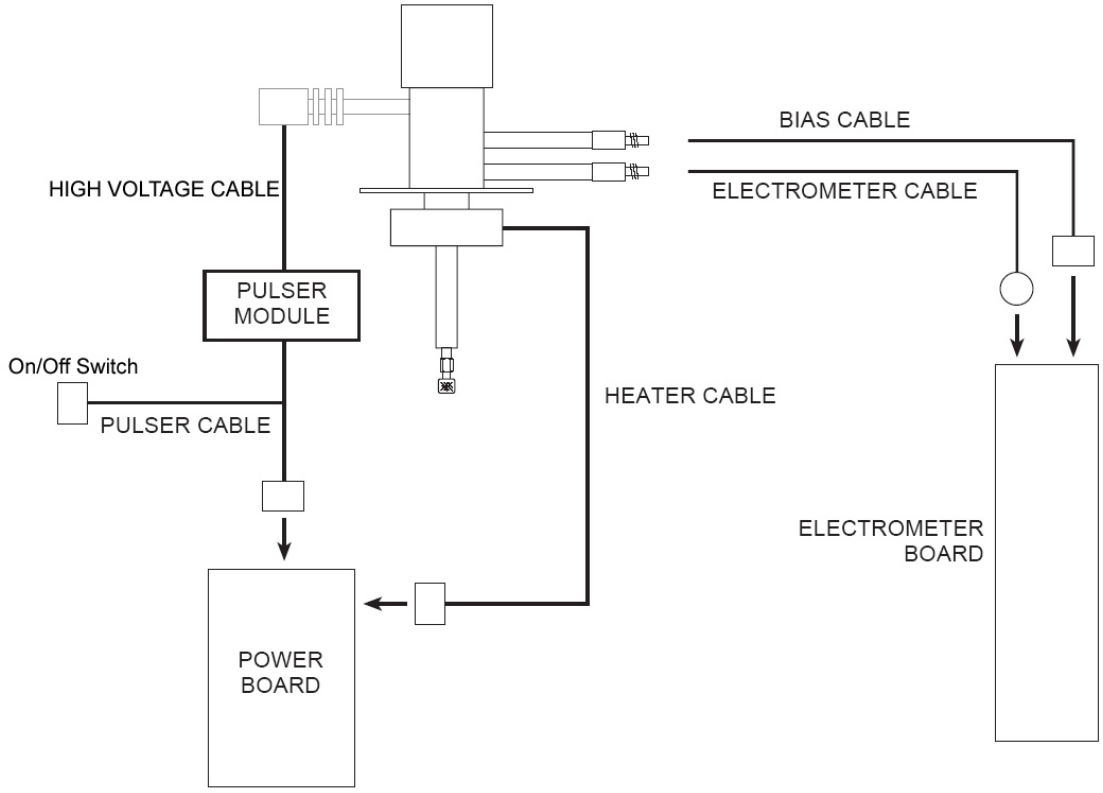
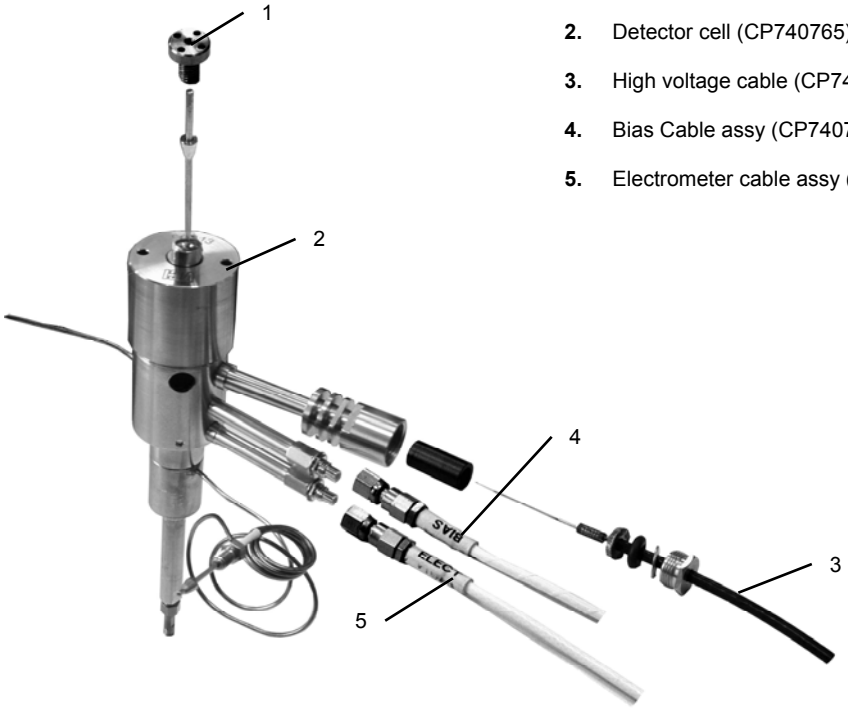


Figure 49: PDHID Cable Overview

Exploded View



1. Ground electrode (CP740764)
   Nut for ground electrode (CP914060)
   Spanner tool (CP740760)
2. Detector cell (CP740765)
3. High voltage cable (CP740763)
4. Bias Cable assy (CP740761)
5. Electrometer cable assy (CP740773)

Figure 50: PDHID Exploded View

Gas Connections

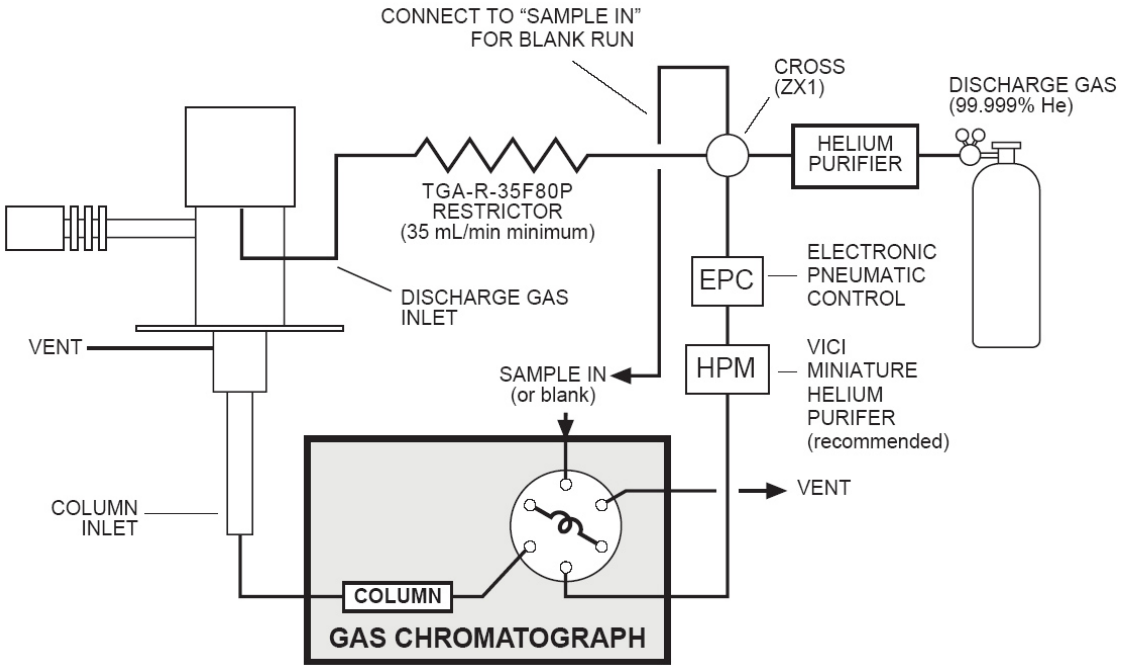


Figure 51: PDHID Gas Connections Overview

Installation

The installation of the PDHID detector is normally done at the factory, field installations are not advised.

General Precautions

- Do not use plastic/polymer or copper tubes for gas handling and interconnections. Use only stainless steel tubing with Valco gold-plated ferrules.
- Do not turn the unit on until helium discharge gas is flowing through the detector.
- Do not shut off or disconnect the discharge gas when the detector is hot, even if the unit is turned off. Turn off the discharge power switch and allow the detector to cool down naturally before disconnecting or shutting off the discharge gas.
- Do not cover the unit with anything which could restrict air circulation.
- Position the controller unit where the mains switch on the rear panel can be reached easily.

CAUTION

Never use leak detecting fluids on any part of the system.

Installing and Purging the Helium Purifier

1. If the pressure regulator has a 1/8" male cone-type outlet port, install the Valco 1/8" external to 1/16" internal reducer (EZR21); if it has a 1/4" male cone-type outlet port, install the Valco 1/4" external to 1/16" internal reducer (EZR41). For other regulator outlet fittings, a wide variety of Valco adapters is available.
2. Remove the cap from the inlet tube of the Valco helium purifier and insert the tube fitting into the 1/16" reducer port. (Keep the outlet tube capped.) Use a 1/4" wrench to turn the nut one-quarter turn past the point where the ferrule first starts to grab the tubing. Do not remove the fitting. When made up properly, it should be leak-tight.
3. Turn the output pressure regulating knob clockwise until the gauge registers 345 kPa (50 psi).
4. Allow 5 minutes for equilibration, then turn the regulating knob all the way counterclockwise.

5. Observe the needle of the output pressure gauge for 15 minutes. There will be a slight initial drop, but if it doesn't move after that, consider that all the connections are tight.

6. If necessary, use an electronic leak detector to locate any leaks. If a leak detector is not available, tighten all the fittings (including the output pressure gauge) and repressurize the system for another test.

7. Uncap the outlet tube of the purifier and purge the system for 15 to 30 minutes at 60 - 80 mL/min to eliminate air from the purifier getter material.

Connecting the Discharge Gas to the Detector

1. If you are supplying the GC from the helium purifier, use the Valco ZX1 cross. (The cross supplies an extra port for a blank helium run.) Otherwise, use one of the Valco 1/16" unions (ZU1) to connect the outlet tube of the purifier to the inlet of the supplied discharge gas.

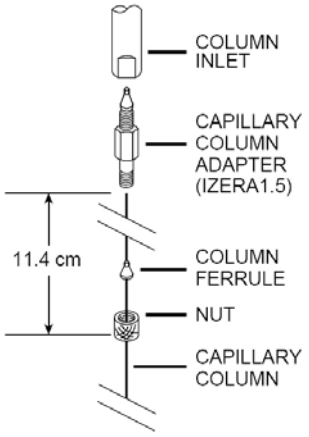
2. Restrictor (TGA-R-35F80P).

3. Connect the outlet end of the restrictor to a flow measuring device. Adjust the helium pressure to 80 psi to obtain a continuous flow of ~35 mL/min.

4. After setting the flow rate, connect the outlet of the restrictor to the discharge gas inlet tube (labeled "INLET") which comes out the side of the detector.

Capillary Column Connection

If the capillary column adapter is installed in the column inlet:



1. Make a mark on the column 11.4 cm from the end.

2. Remove the knurled nut and plug from the capillary column adapter in the column inlet at the bottom of the detector. Slide the nut over the end of the column, followed by the appropriate column ferrule (FS.4 or FS.5, or ZF.5V for megabore).

3. Seat the ferrule in the detail of the column adapter and begin sliding the column through the capillary column adapter and into the column inlet.

4. Get the nut started on the threads and tighten it until you feel it contact the ferrule, then back off half a turn. Slide the column into the column inlet until the mark is flush with the surface of the knurled nut and secure the column in the adapter by tightening the knurled nut finger tight only.

If the capillary column adapter has been removed, reinstall it:

1. Unscrew the liner as far as it will go, then screw the fitting body into the column inlet fingertight.

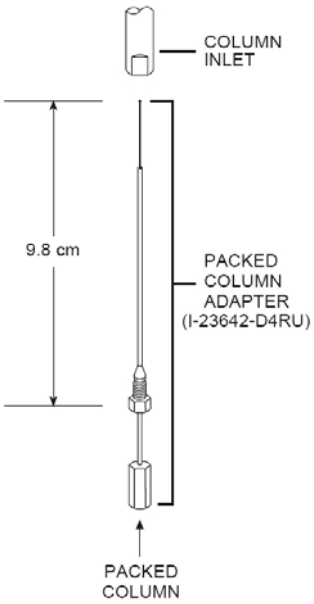
2. While using a 1/8" wrench to prevent rotation of the liner (the part with the seat for the column ferrule), use a 1/4" wrench to tighten the body of the adapter until the ferrule has sealed. The liner will deform if it rotates.

3. Proceed to Step 1 above.

Packed Column Connection

To prevent detector contamination, we strongly recommend disconnecting the column from the detector during column bakeout procedures.

The PDHID is optimized for packed columns. The column tubing must be thoroughly cleaned and baked before the column is packed. Even when the best care is taken in column tubing cleaning and in the support and stationary phase selection, a new column will often bleed compounds, resulting in a considerable increase in the detector baseline. This initial bleed will usually be reduced to acceptable levels after the column is conditioned with clean carrier gas flow for several hours at the recommended bakeout temperature.



1. Loosen and remove the knurled nut and plug of the capillary column adapter, (or remove the column ferrule and the column if one has been installed).

2. Use a 1/8" wrench to hold the liner that part of the adapter in which the column ferrule sits. While the 1/8" wrench keeps the liner from rotating, use a 1/4" wrench on the fitting body to loosen the adapter 1/2 turn.

3. Set aside the 1/8" wrench and completely remove the adapter from the column inlet.

4. Screw the packed column adapter into the column inlet by hand. Exercise caution, as the tip of the adapter is very fragile. Then tighten the adapter with a 1/4" wrench, using an additional wrench on the flats of the column inlet to support the detector.

5. Connect the 1/8" column to the packed column adapter with the EZRU21 reducing union supplied in the fittings kit.

Testing for Leaks

It is critical for the system to be leak-tight and an additional check at this point can save many headaches later on. To test for leaks:

1. Cap the tube and pressurize the entire system with helium to 138 kPa (20 psi).
2. If the system does not hold pressure, check all the fittings with an electronic helium leak detector. DO NOT use leak detecting liquids.
3. Tighten fittings as required.

THERMIONIC SPECIFIC DETECTOR (TSD)

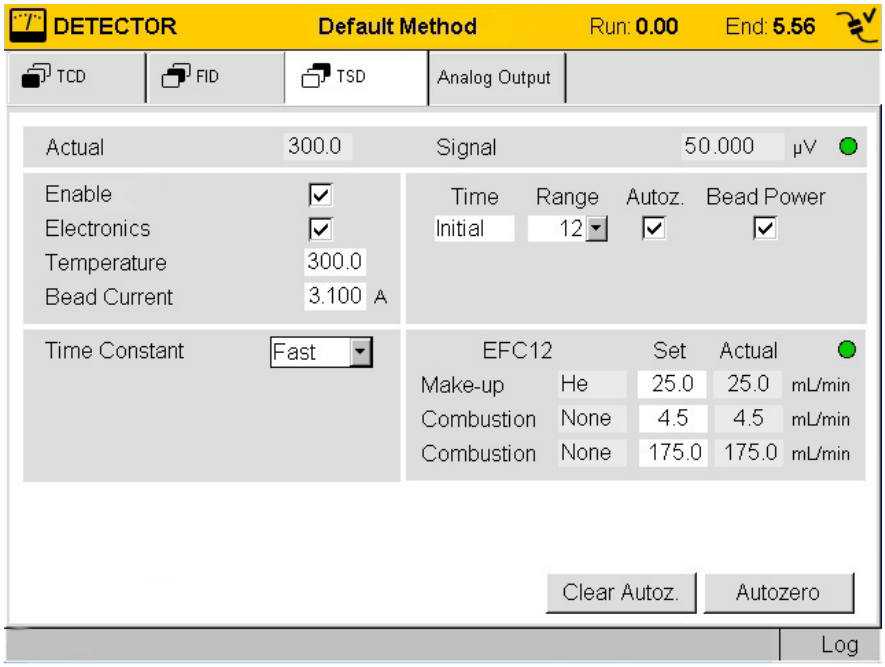
The Thermionic Specific Detector (TSD) is also known as *Nitrogen Phosphorus Detector* is a very sensitive but specific detector that responds almost exclusively to nitrogen and phosphorous compounds. It is based on the flame ionization detector but differs in that it contains a rubidium silicate (glass) bead situated in a heater coil, a little distance from the hydrogen flame. If the detector is to respond to both nitrogen and phosphorous then the hydrogen flow should be minimal so that the gas does not ignite at the jet. If the detector is to respond to phosphorous only, a large flow of hydrogen is used which is burn at the jet. The heated bead emits electrons by thermionic emission. These electrons are collected under a potential of a few volts by an appropriately placed anode and provides a background current. When a solute containing nitrogen or phosphorous is eluted from the column, the partially combusted nitrogen and phosphorous materials are adsorbed on the surface of the bead. The adsorbed material reduces the work function of the surface and, as consequence, the emission of electrons is increased which raises the current collected at the electrode. The sensitivity of the detector to phosphorous is about 10-12 gram per mL and for nitrogen about 10-11 gram per mL at a signal to nose ratio of 2. The bead has a finite life and needs regular replacement. These instructions refer to the installation and operation of the Thermionic Specific Detector (TSD) on the 450-GC. The TSD detector tower is installed on a 450-GC detector oven. The TSD Electrometer Board is installed in the GC electronics cabinet on the left side of the instrument. The TSD is controlled through the keyboard on the front panel of the GC. Gas flows are controlled at the pneumatics panel of the GC, or in the Adjustments section of the method if Detector EFC is installed.

Initial Set-Up

To set up the TSD for operation, proceed as follows. Note that when the 450-GC is first powered up all heated zones with the exception of the column oven are powered off and all detector electronics are turned off.

1. Refer to the Pre-Installation Instructions for additional information on gas supply requirements.

2. Press the DETECTOR key on the 450-GC Display/Keyboard to display the DETECTOR screen. The TSD should be listed in location Front (), Middle () or Rear (). If your 450-GC is equipped with detector EFC, verify the makeup gas you will be using with your TSD.



3. Install a chromatographic column in the GC (refer to the Column installation information on page 23). If the analytical column is not well conditioned, use a no-hole ferrule in the detector column nut while conditioning the column and leave the column end loose in the oven. If the analytical column is well conditioned, follow the usual procedure for column installation.

4. Condition the TSD bead. The following bead conditioning and optimization procedures assume use of a short, non-polar column such as the Varian test column, 03-912300-30 (packed, COLUMN, 1/8" X 50 CM 5%, OV101) or CP887098 (capillary CP-Sil 5 CB, 4M X .32). If you use a different type of column, you should adjust conditions accordingly.

Operation and Operating Recommendations

TSD operation requires hydrogen, air+make-up and either nitrogen[a] or helium carrier gas. The amount of hydrogen relative to the other gases will affect nitrogen-to-carbon and phosphorus-to-carbon selectivity of the detector. The nitrogen-to-phosphorus selectivity also may vary somewhat but the detector cannot be tuned to respond to only nitrogen or only phosphorus.

When power is first applied to the TSD, the bead may require 2 to 3 minutes to re-light, i.e. stabilize the bead surface temperature and gas phase reactions. If the bead does not re-light, check for other errors and review the bead conditioning procedure and the bead optimization procedure.

Bead Conditioning

This TSD bead probe requires additional conditioning to perfect detector performance. Follow these recommended procedures to ensure maximum lifetime and reliability of your TSD bead. Failure to follow these procedures may result in poor performance and reduce TSD bead probe lifetime.

Each bead probe will have a particular bead current (i.e. bead surface temperature) at which its gas phase chemistry will initiate. In order to operate the detector, it is necessary to allow the bead to reach this ignition temperature in a controlled manner. Attempting to accelerate this procedure may decrease the lifetime of the TSD bead probe.

1. Check the position of the TSD bead in the detector tower. The TSD bead must NOT make contact with the TSD collector screen. The bead should be located in the center of the collector, with the collector positioned so that both edges of the screen are equidistant from the bead.

2. Install a short, non-polar column such as the Varian test column, 03-912300-30 (packed, COLUMN, 1/8" X 50 CM 5%, OV101) or CP887098 (capillary CP-Sil 5 CB, 4M X .32). If you use a different type of column, you should adjust conditions accordingly.

[a] Nitrogen will prolong the bead life time and performance.

3. Set gas flow rate (should be measured at the flame tip):

Air	175 mL/min.
Hydrogen	4.0 mL/min.
Carrier gas (+ make-up)	30 mL/min.

4. Set temperatures:

Packed Column	175 °C (isothermal).
Capillary Column	80 °C (1 min), 20 °C/min to 200 °C (2 min).
Injector	230 °C.
Detector	300 °C.

5. Set the initial Column oven temperature. Set the desired Injector temperature to which your TSD column is connected. Check that the Injector oven power is ON. Detector temperature is set by pressing the DETECTOR key, selecting the TSD, setting the temperature to 300 °C and turning the Oven Power ON. Leave the Electronics and beadcurrent OFF at this point. Set the detector to range 12 and AUTOZ. unchecked.

6. Allow the TSD detector to reach temperature and allow the bead to thermally equilibrate for at least 15 minutes prior to turning on the electronics.

7. Set the initial Autozero OFF. Set the bead current to 3.100 A. Turn on the Bead Power. Allow the bead to condition for at least 15 minutes.

8. Increase the bead current in steps of 0.100 Ampere, waiting at least 15 minutes for the bead to equilibrate between increases. When increasing the bead current causes the TSD Signal to go above 64 mV, the bead has reached its ignition point. Allow the bead current to remain at this setting for a minimum of 12 hours (preferably overnight). This completes the initial conditioning of the TSD bead probe. Now optimization of the bead probe for N/C selectivity is required.

The Clear Autozero button removes the Autozero offset so that you can observe the detector background signal.

Optimization of the TSD Using the Varian Test Sample

The sensitivity of the TSD is highly dependent on bead temperature and the selectivity is dependent on hydrogen flow. In addition, there are slight variations from bead to bead and a bead changes slowly as it ages. Therefore, optimum operating conditions for a given TSD cannot be specified exactly; the user should determine optimum operating conditions for the application and TSD bead.

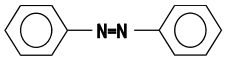
The detector responds to both nitrogen and phosphorus. It is not possible to significantly change the relative response of nitrogen to phosphorus.

Determining Optimum Hydrogen Flow

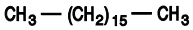
Although a nominal hydrogen flow of 4.0 mL/min is suggested, the nitrogen-to-carbon selectivity versus hydrogen flow often differs slightly from bead to bead. Consequently, for each new bead it is useful to chromatograph the detector test sample at several values of hydrogen flow (from 4 to 5.5 mL/min) to determine the best hydrogen flow for that bead.

1. Zero the TSD signal. Inject 1 µL of TSD test sample (82-005048-04). A normal response will result in the bead quenching during the solvent peak (i.e. a negative deflection of the pen will occur) and a positive response for the azobenzene, heptadecane, methyl parathion and malathion peaks. Adjust the detector range to keep the peaks on scale.
2. If the azobenzene peak (see the appropriate chromatogram below) is less than 3 times the height of the heptadecane peak, decrease the hydrogen flow rate (in small steps) and re-inject the sample. If injection of the test sample results in quenching by the solvent peak and the bead fails to re-ignite, simply increase the bead current slowly (use small steps), with intervening pauses of ~15 minutes, until the bead re-ignites. Then repeat step 1.
3. Over the next few days, the background current of the bead will decrease and the sensitivity will also decrease to a steady level. At this point the sensitivity of the bead will remain relatively constant, with low baseline drift and low noise.

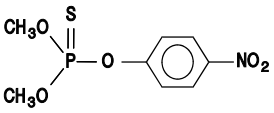
1. Azobenzene:
   2 ng Azobenzene contains 310 pg nitrogen



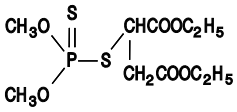
2. Heptadecane:
   4 μg heptadecane contains 3.4 μg carbon



3. Methylparathion:



4. Malathion:
   4 ng Malathion contains 380 pg phosphorus



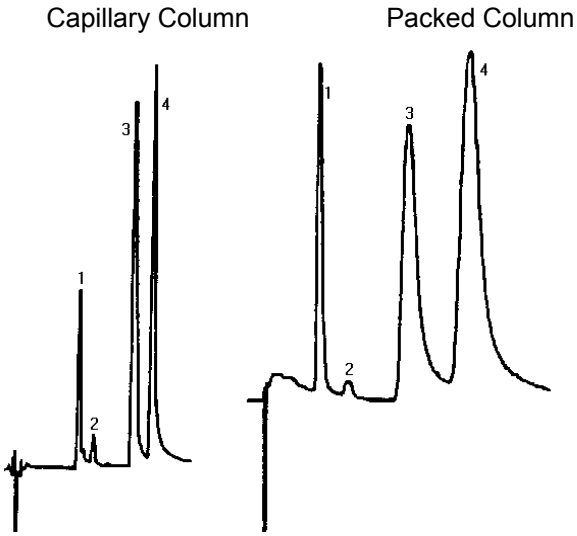


Figure 52: Structure of Components in the TSD Test Sample & Chromatogram examples

Maintaining Optimum Conditions

If the detector is used daily, the power to the bead may be left on when not in use at 2.4 to 2.6 Amps. As the bead ages, it will require a higher current to maintain a given bead sensitivity. Therefore, detector background signal should be checked every few days. Eventually sensitivity will be lost and it will be necessary to replace the bead (03-906074-01 conditioned or 03-906074-00 **UN**conditioned). The hydrogen flow does not need to be changed during the lifetime of a bead.

Column Bleed

To minimize contamination of the detector by column bleed, all new columns should be conditioned before they are connected to the detector. Condition new columns for a minimum of 24 hours at 40 °C (or 2 hours at maximum analysis top) below the maximum temperature of the column packing liquid phase.

Since the TSD detects compounds containing nitrogen or phosphorus, column packing materials containing these elements should be avoided. Common liquid phases containing nitrogen are OV-225, OV-275, FFAP, XE-60 and TCEP. If you must use such columns, it is important that they be well conditioned and operated at as low a temperature as possible in order to minimize column bleed. High column bleed produces high background signals, making it difficult or impossible to set the bead current and can result in the occurrence of negative peaks in the chromatogram.

Solvent Considerations

When the TSD is used for trace level analysis, only solvents free of nitrogen, phosphorus, sulfur, or halogenated compounds should be used. Depending on the volatility of the impurity in a solvent, it can produce excessive solvent tailing or extraneous chromatographic peaks.

Chlorinated Solvents

Chlorinated solvents can be used with the TSD. However, these solvents generally cause an abrupt increase in both detector background signal and sample response. These effects appear to be associated with a temporary adsorption of chlorinated species onto the surface of the hot alkali-ceramic bead. Repetitive injections of samples in chlorinated solvents yield constant responses. However, once the injection of chlorinated solvents is stopped, the background and sample response will decay back to the response levels that existed before the initial injection of the chlorinated solvent. Therefore, when chlorinated solvents are used, calibration standards should be run frequently.

You can minimize the effect of chlorinated solvents on the bead by turning off bead current during the time that the solvent elutes, using the time programming table of the detector method. The total time that the bead is turned off should be less than 2 minutes in order to minimize thermal re-equilibration times of the detector. (Any peaks eluting during this off time will not be measured.)

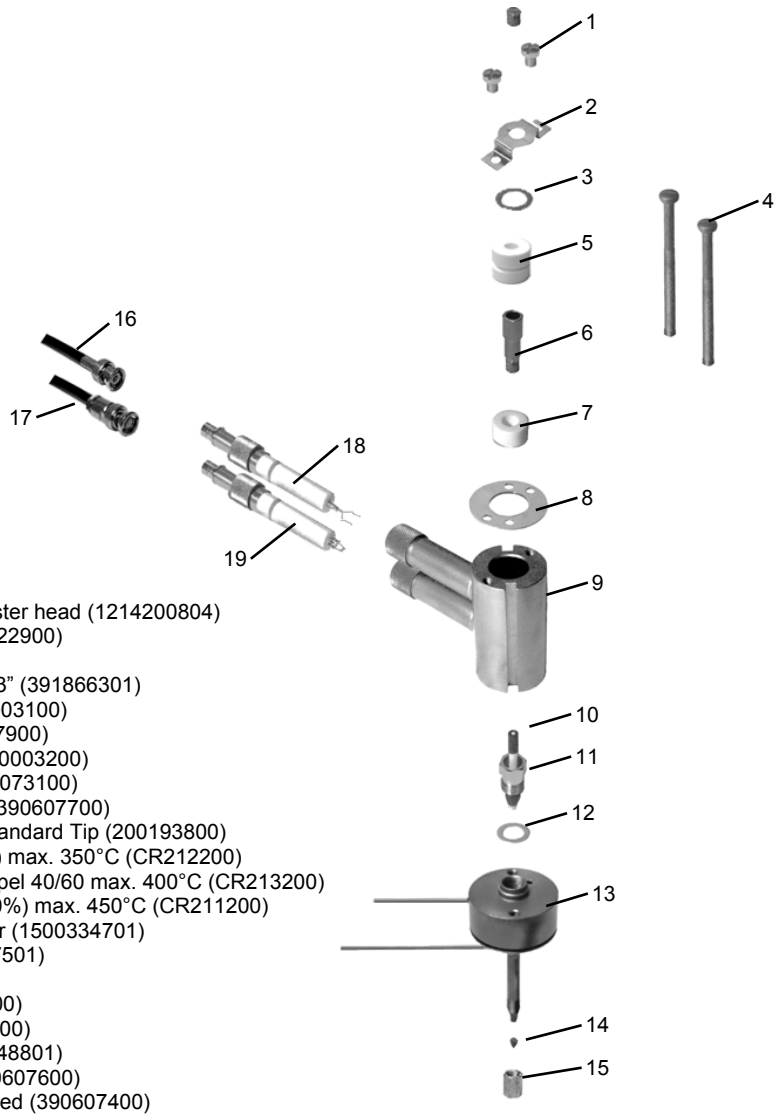
Effect of Silylation Reagents

Silylation reagents affect the TSD in 2 ways. First, such reagents sometimes contain nitrogen which will cause tailing of the solvent peaks. Second, decomposition of the reagent on the hot bead produces SiO_2 which deposits on the bead surface causing loss of response. Generally, SiO_2 deposits can be removed by cleaning the bead and usually produce no permanent damage to the bead.

TSD Assembly

It may be necessary to remove the TSD detector components for inspection, cleaning, parts replacement, or installation of another detector. Disassembly/re-assembly instructions are detailed below for both the TSD and the detector oven.

Exploded view



1 Screws, 8-32 x ¼" Fillister head (1214200804)
2 TSD Top Clamp (800022900)
3 O-ring (2740928202)
4 Tower Screws, 8-32 x 3" (391866301)
5 Upper Insulator (2100003100)
6 Collector Tube (390607900)
7 Ceramic Insulator (2100003200)
8 Tower Top Plate (3400073100)
9 Detector Tower Body (390607700)
10 Flame Tip, 0.010 in. Standard Tip (200193800)
11 Ferrule: Vespel (100%) max. 350°C (CR212200)
Graphite/Vespel 40/60 max. 400°C (CR213200)
Graphite (100%) max. 450°C (CR211200)
12 Aluminum Seal Washer (1500334701)
13 Detector Base (392547501)
14 Column Ferrule
15 Column Nut (394955100)
16 Signal Cable (200187700)
17 TSD bead cable (392548801)
18 TSD Signal Probe (390607600)
19 TSD bead, unconditioned (390607400)
, conditioned (390607401)
--- Flow Tube (200187600)

Figure 53: TSD Exploded View

TSD disassembly

1. The TSD is mounted on the ion detector base in the detector oven.
2. Turn the TSD Oven Power and Bead Power OFF. Allow the detector to cool to ambient temperature.
3. Turn off the main GC gas supplies to the detector at the pneumatics panel.
4. Disconnect the signal and the igniter cables from the TSD tower.
5. Remove the 2 tower mounting screws and lift the detector tower vertically from the detector base until clear of the flame tip. Remove the 2 screws from the top clamp.

Use clean cotton gloves or tweezers when handling internal parts of the TSD.

CAUTION

Avoid contamination of ceramic insulators and probes. If the detector is not completely cool, use a metal tool (such as tweezers or a hooked wire) to remove parts from the tower assembly.

6. Remove the upper insulator.
7. Remove the signal probe. There may be some resistance to removal, due to the O-ring seals. Do not rotate probe as it is withdrawn from electrical contacts in tower.
8. Remove the collector and the lower insulator with tweezers.
9. Remove the bead probe. There may be some resistance to removal, due to the O-ring seals. Do not rotate probe as it is withdrawn from electrical contacts in tower.
10. Carefully remove the flame tip assembly from the detector base.
11. Remove and discard the aluminum seal washer from detector base. In some cases this washer may remain in the detector tower. If so, be sure to remove and discard this washer so that the detector is not accidentally reassembled with both the old washer and a new washer.

TSD Re-assembly

Refer Figure 53 for re-assembly orientation.

1. Re-assembly is the reverse of removal.

2. Install the flame tip in the detector base. This is a 0.020-inch ID ceramic flame tip. The TSD and FID flame tips are identical. DO NOT use a 0.010-inch flame tip for the TSD.
   a. If you are installing the flame tip assembly with a used Vespel® ferrule, tighten the assembly finger-tight plus an extra 1/6-turn.
   b. If you are installing the flame tip assembly with a new Vespel ferrule, tighten about 1/3-turn past finger-tight.

3. The Vespel ferrule supplied with the GC has a maximum temperature limit of 350°C. If it is ever necessary to operate the detector above 350 °C, replace the Vespel ferrule with a graphite ferrule.

4. Install a new aluminum seal washer onto the shoulder of the detector base.

For a reliable tower seal, use a new aluminum washer each time the detector is installed.

5. Measuring the nominal 4.0 mL/min H_2 flow for the TSD may be simplified at this time by attaching a tube to the flame tip and then connecting the tube to a flowmeter. Make sure the makeup gas is turned of and adjust the hydrogen flow.

6. Replace the detector tower on the detector base and secure with the 2 screws. Tighten screws alternately as tower is tightened into place.

CAUTION

Handle the ceramic insulators and probes with tweezers to avoid contamination. Use care when handling to make sure bead probe is not bent or dropped, as the ceramic bead on the probe is fragile.

7. Carefully insert bead probe into lower arm of detector tower. Align probe key with tower arm slot.

8. Secure bead probe by tightening knurled nut.

9. Insert lower insulator with shoulder down into detector tower, then insert collector tube with grid down. Make sure the punch mark on the top edge of the collector is toward the probe arm.

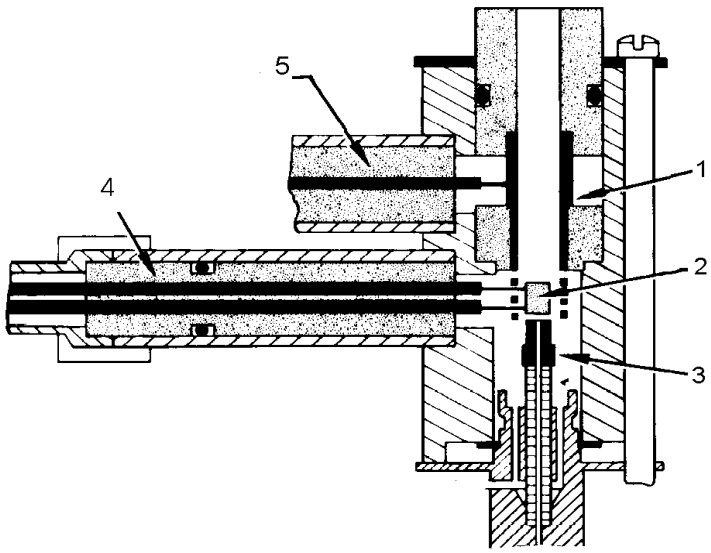
10. Insert signal probe into upper arm of detector tower. Carefully align the spring clip with the collector. Be sure the key on the probe and the tower arm are aligned. Secure signal probe by tightening knurled nut.

11. Assemble the upper insulator and top clamp over the collector. The O-ring will provide slight resistance to complete insertion. Take care not to tear the O-ring or the tower will not seal properly.

12. Look down the tower assembly to verify that the bead does not touch the collector. Carefully adjust the collector as necessary.

13. Assemble the top clamp to the tower and secure with the 2 screws.

14. Connect the signal cable to the signal probe (top) and the igniter cable to the bead probe (bottom). If cables were totally disconnected from the GC, reconnect signal cable and the bead current supply cable at the top of the TSD Electrometer Board.



1 Collector with Screen Bottom
2 Ceramic Bead with Heater Coil
3 Flame Tip
4 Bead Probe
5 Signal Probe

Figure 54: TSD Cross-Sectional View

MAINTENANCE

Cleaning / Maintenance

When the TSD exhibits low sensitivity or high background noise, cleaning is indicated. Visual inspection may also indicate a need for cleaning. The ceramic bead, collector, or flame tip are all parts that are deposit prone, depending on the type of samples being used. Clean these parts as indicated in the following paragraphs.

Always handle detector components with tweezers to avoid contamination.

Bead Cleaning

Non-volatile deposits can be removed from the bead by gently scraping the bead surface with a sharp tool or abrasive paper. The bead should be supported while being cleaned to prevent bending the lead and cracking the ceramic coating on the leads.

Flame Tip and Internal Parts Cleaning

Follow the steps below to clean the flame tip and internal parts.

Use proper eye and skin protection. Methanol and acetone are toxic and flammable chemicals. Exercise appropriate precautions when these chemicals are used.

1. Remove the detector and internal parts. Handle internal parts with tweezers and place on a clean, uncontaminated surface.
2. Clean deposits from the surface of the collector with water or abrasive paper.
3. Remove deposits which form on top of the flame tip by scraping. If flame tip is plugged, clear by inserting wire (approximately 0.012 inch diameter) into flame tip orifice.

4. Flush cleaned components with methanol and air dry.

Replace the Flame-Tip Ferrule

After a period of prolonged use, the flame-tip ferrule may deteriorate and crack. As a consequence, a leak may develop around the base of the flame tip assembly that causes unstable baseline noise and reduced sensitivity. In this situation, or when the detector is to be operated at temperatures above 400 °C, you must replace the ferrule. Be sure to select the proper ferrule for the application. The Vespel/graphite ferrule has a maximum temperature limit of 400 °C see Figure 53 on page 313.

Test Chromatogram

For an FID test chromatogram see section Detector Test Chromatograms, on page 421.

THERMAL CONDUCTIVITY DETECTOR (TCD)

A TCD detector consists of an electrically-heated wire or thermistor. The temperature of the sensing element depends on the thermal conductivity of the gas flowing around it. Changes in thermal conductivity, such as when organic molecules displace some of the carrier gas, cause a temperature rise in the element which is sensed as a change in resistance. The TCD is not as sensitive as other detectors but it is non-specific and non-destructive.

The Thermal Conductivity Detector (TCD) is mounted directly above the column oven on the 450-GC. The TCD electronic Board is installed in the electronics cabinet on the left side of the instrument.

TCD operating parameters are set at the 450-GC Display/Keyboard: These include carrier gas type, detector temperature, range, temperature limit and filament temperature. The gas flows are set and controlled either from the 450-GC Display/Keyboard (if detector EFC is installed) or at the pneumatics panel.

Before Operating the TCD

Do not operate the TCD until you have connected carrier gas to both inlets of the TCD. The TCD accommodates 2 columns (a column for the reference cell and a column for the detector cell) or a column and a reference line. The columns can be packed or capillary columns and you can combine a capillary column with a packed column. To avoid contaminating the detector, condition all columns before you connect them to the TCD.

Initial Set-Up

To set up the TCD, proceed as follows:

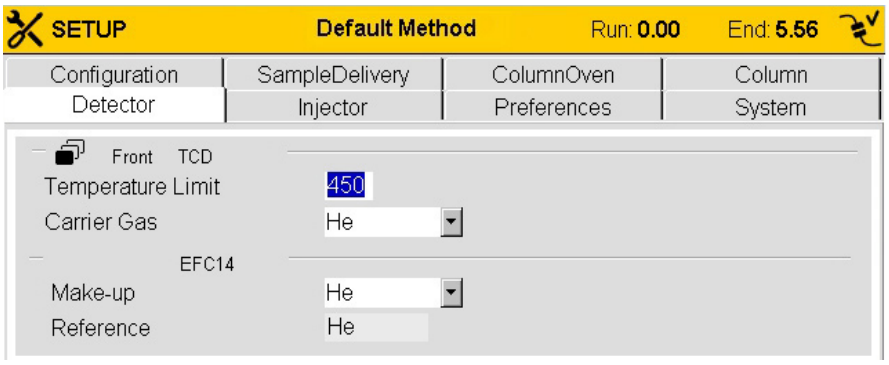
Note that the TCD is a 2 channel detector and requires both a sample flow of carrier gas and a reference flow of the same gas. Either channel can be used as the sample stream but there must always be a reference stream.

There are TCD parameters which need to be set before using the TCD for analysis. These are the choice of carrier, make-up and reference gas and the filament temperature limit.

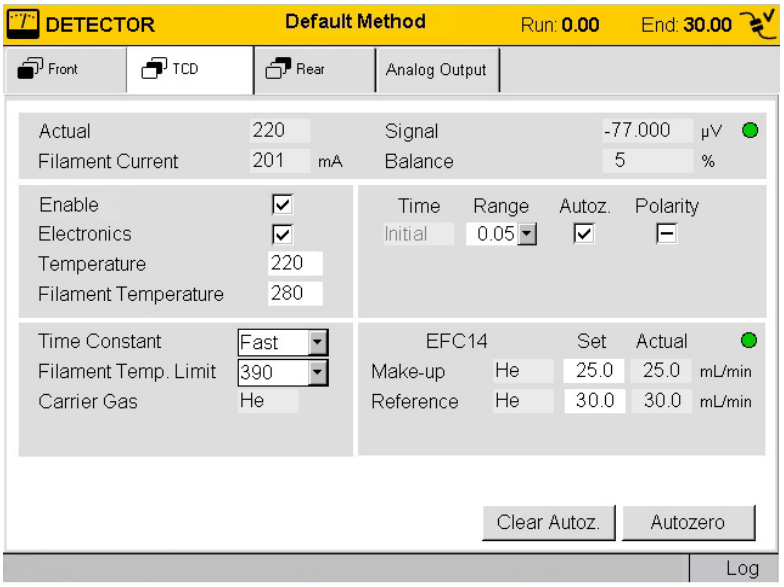
1. Refer to the Pre-installation Instructions for additional information on gas supply requirements.

2. Check the gas supply pressures. Flow controllers with turn dials are calibrated for Helium at 80 psig. Recalibrate the flow controllers if you use a different supply pressure or carrier gas.

3. Press the SETUP key on the 450-GC Display/Keyboard. Select the TCD by location. Select the carrier, Make-up gas and TCD oven temperature limit. Press OK.



4. Press the DETECTOR key on the 450-GC Display/Keyboard. The TCD should be listed in Position Front (), Middle () or Rear ().



5. Set the detector and filament temperature limit and time-constant. To protect the filaments, always operate the detector with the 390 °C limit selected unless you require the maximum dynamic range available to the TCD.

6. Establish carrier gas flow through the Sample (right) side of the TCD and an equal Reference gas flow through the Reference (left) side of the detector. If you are using a capillary column, the total of column flow and makeup flow should equal the Reference gas flow. Perform a Leak Check on both the Sample and Reference pneumatics before proceeding to apply power to the TCD filaments. Even a small leak can supply enough oxygen to cause filament oxidation and consequent baseline drift.

TCD Adjustments

With prolonged use, the filaments slowly oxidize and their resistances increase. At some point this will cause the largest peaks in your chromatogram to become flat topped, indicating that the filament protection software has been activated. (Note that if the TCD signal is sufficiently large the output may go to zero and become unresponsive. If this occurs, adjust your injection site and conditions to avoid over range conditions.) To adjust for this increase in resistance, set the Filament Temperature Limit to 490 °C, even for filament temperature setpoints below 390 °C.
The TCD filament is automatically protected in the GC. The GC turns off the filament current after 4 minutes if it detects that the carrier gas flow has stopped or that the filament current is low. Also, the GC turns off filament current when it detects that carrier gas is flowing through one side of the detector cell but not through the other (e.g., a septum or column is not installed, or a leak exists in one cell and not the other).
If you select Helium or H_2 as the carrier gas, the GC operates in the standard way to protect the TCD filament. However, because the filament protection feature in the GC operates through the detection of air (or N_2) in the detector cells, the filament protection feature is disabled when you use nitrogen or argon as the carrier gas.

Set the Filament Temperature Limit to 390 or 490 °C, according to the highest filament temperature setpoint required. With the Limit set to 390 °C, the maximum filament temperature is limited to approximately 450 °C. The temperature limit is approximately 550 °C at the 490 °C setting. The 450 °C limit protects filaments from oxidation when exposed to air over an extended period of time. However, at the 550 °C temperature limit the filament can degrade after only a few minutes.

If you select N_2 / Ar as the carrier gas, the filament protection feature is disabled. Because the GC does not turn off the filament current, the TCD filaments can rapidly oxidize if you operate the TCD filament temperature above 390 °C without carrier gas flowing through the TCD cells. If you plan to operate the TCD after the carrier gas has been turned off, purge the air from the TCD cells with carrier gas for 5 minutes before operating the TCD.

The TCD can be used with 2 different types of EFC. The EFC14 provides make-up flow to one channel of the TCD and reference flow to the other channel for Helium, Argon or Nitrogen as carrier gas. The EFC16 is used for operating the TCD with hydrogen as carrier gas.

Operation

To operate, the TCD requires only carrier gas. Helium, Argon or Hydrogen (only with special EFC16) are recommended as carrier gases. You can use nitrogen as a carrier gas, but you may lose some sensitivity and see an increase in the detector noise.

WARNING:
FIRE HAZARD

If hydrogen is used as a carrier gas, vent the hydrogen from the TCD to a safe place. Hydrogen is very flammable.

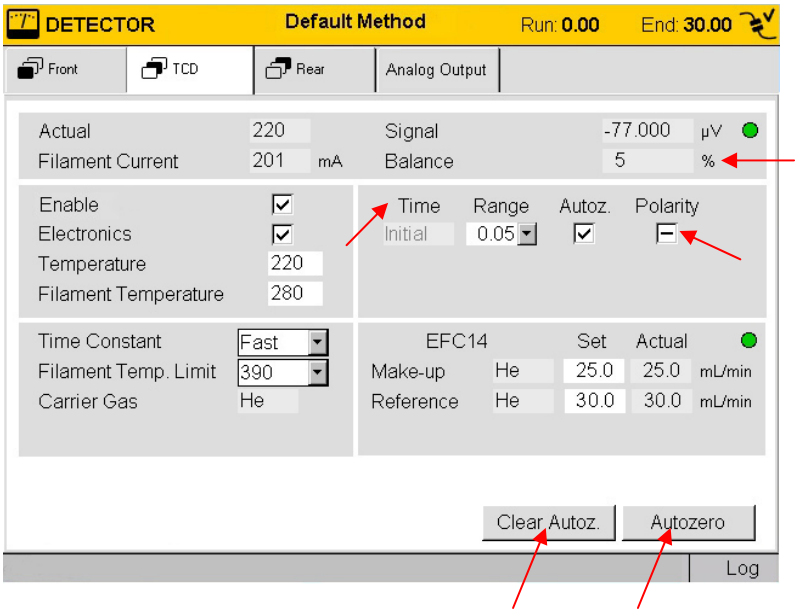
Assume that the analytical column is attached to the right side of the TCD and the reference column or reference gas supply to the left side of the TCD. However, you can configure the GC with the analytical column connected to the left side of the TCD, simply reverse the TCD polarity.

If you plan to operate the TCD after the carrier gas has been turned off for an extended period of time, turn on the carrier gas for 5 minutes before turning on the filament current to purge air from the system. This prevents oxidation of the TCD filaments.

At the factory, the carrier gas flow rate for TCD is set at 30 mL/min. (Carrier gas: He) through both sides of the detector. The carrier gas flow rate is adjusted at the pneumatics cabinet or the Detector page of the display and measured at the 2 exit ports on the TCD. Once the carrier gas is flowing through the TCD, set up the TCD for operation as follows:

1. Set the TCD oven to the desired temperature and allow it to come to the setpoint. Usually, set the TCD temperature at least 20 °C higher than the maximum temperature the column oven reaches in your analysis.
2. Set TCD filament temperature. A filament temperature 50°C above the detector temperature should provide a good starting point for most applications. After the TCD oven reached its set temperature, turn the TCD Electronics ON to begin heating the filaments.
3. Allow system to equilibrate 10 to 15 minutes.
4. Monitor the filament current through the detector status display. Initially, the current is higher than the final operating current. Gradually, however, the current decreases as the detector thermally equilibrates. For best performance of the detector, do not permit the filament temperature to exceed the detector temperature by more than 200 °C. Generally, use the lowest detector current possible for the sensitivity your application requires.

5. Un-check AutoZ. (Autozero OFF). Press the Autozero button to set the initial signal to 0. Monitor the detector signal. A continuous drift, either positive or negative, may indicate an air leak in either the carrier or reference gas stream.
6. Note that it is normal for the TCD baseline to drift until the detector thermally equilibrates. The time it takes to reach equilibrium always exceeds the time it takes for the detector oven to reach its temperature. When the baseline is stable, you can begin your analysis. Check the AutoZ. (Autozero ON). Autozero is automatically disabled when the GC method is in RUN.



The Autozero button applies an immediate zeroing of the detector signal.
The Clear Autoz. button will clear the autozero.
Time box allows time programming events such as range, autozero and polarity changes.

Initial Autozero = YES allows the system to balance the bridge automatically before each run for maximum dynamic range. The current status of the balance control, from -99 to +99%, is shown on the display.

A continuous drift in the balance value over time may indicate you have an air leak which is causing one set of filaments to oxidize, changing their resistance and unbalancing the detector bridge.

If you inject a standard or sample into the reference side of the TCD, peaks appear in the negative direction. If you want to integrate or process these peaks with a data system, you must convert the polarity of the peaks from negative to positive. Use the checkbox Polarity.
Use the time program when you would like the polarity to be automatically reversed during a chromatographic run.

Range Setting

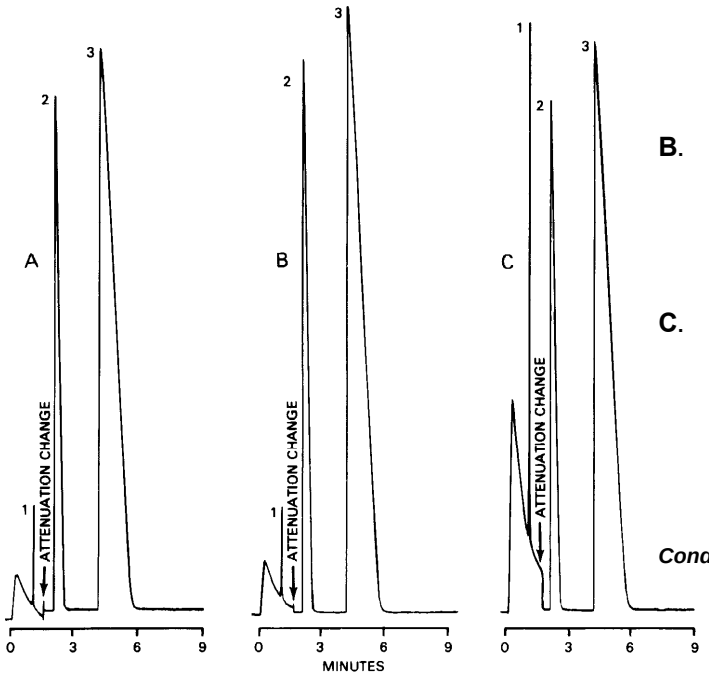
Use the 0.05 mV range in the following cases:

- Low currents (100-150 mA).
- High Currents (300 mA).
- Output Signal Fed to Computer or Integrator.

Low Currents (100-150 mA)

Analyses that required a high filament current and a less sensitive range can often be performed also better at lower currents and a more sensitive range. For example, a filament current of 120 mA and a range of 0.05 mV yields about the same output signal as a filament current of 300 mA and a range of 0.5 mV. At the lower current, the detector noise is lower, the stability is higher and the filament lifetime is extended. Thus, Varian recommends that, whenever possible, you operate the TCD at the lowest practical filament current.

The chromatograms in Figure 55 illustrate the high sensitivity and detectivity (18 ppm of Neon in air) that is possible with the TCD detector. Also, a comparison of chromatogram A with chromatogram B shows that with the use of the bridge output signal amplifier, you can obtain the same analytical results at half the filament current, thereby extending the lifetime of the TCD filament. The Neon peak in chromatogram C illustrates how you can use a high filament current (300 mA) with a high amplification setting to increase the effective sensitivity of the TCD.



A.

Filament current: 290 mA

Filament temperature: 300 °C

At 1.5 minutes, range and attenuation changes from 2 x 0.5 mV to 128 x 5.0 mV.

B.

Filament current: 151 mA

Filament temperature: 160 °C

At 1.5 minutes, range and attenuation changes from 2 x 0.05 mV to 128 x 0.5 mV.

C.

Filament current: 299 mA

Filament temperature: 300 °C

At 1.5 minutes, range and attenuation changes from 4 x 0.05 mV to 128 x 5.0 mV.

Conditions: Carrier gas: He
Sample: Air, 2 mL
Column: 10 ft. molecular sieve 5A 30/60 mesh at 50 °C
Injection: Gas sample valve

TCD oven temperature: 120 °C

Figure 55: TCD Sensitivity and Amplification

High Currents (300 mA)

To obtain the highest possible sensitivity, operate the TCD at the 0.05 mV range with a high filament current. In this configuration, TCD noise is primarily noise derived from filament vibration and fluctuations in carrier gas flow through the detector. However, at high filament currents, the detector is very susceptible to air leaks into the gas lines, improperly conditioned columns, imbalanced reference and analytical flow rates, or impure carrier gas supply, which cause the baseline to drift. Baseline drift from any of these sources generally increases as the absolute filament temperature increases. Therefore, operate the TCD with as low a filament current as practical for your application.

Also, operate the TCD with the lowest filament and detector temperatures appropriate. If possible, avoid operating the TCD with a filament temperature greater than 390 °C. To operate the TCD with filament temperatures between 390 and 490 °C, set the Filament Temperature Limit to 490 °C. See Figure 55 for an example of the performance of the TCD at a high current.

Calculate Detector Sensitivity

To calculate the sensitivity of the TCD, proceed as follows:

EXAMPLE 1: Known Sample Weight

$$S = \frac{P \times Fc}{W}$$

Where:

S = Sensitivity in mV x mL/mg.
P = Integrated peak area in mV x min.
W = Weight of sample in carrier gas in mg.

F_C = Carrier gas flow in mL/min corrected to detector temp

$$Fc = Fo \times \left(\frac{Td}{Ta} \times \left(1 - \frac{Pw}{Pa} \right) \right)$$

Where:

F_O = Flow rate measured at detector outlet (at ambient temperature) in mL/min.
T_D = Detector temperature in K (Kelvin).
T_A = Ambient temperature in K (Kelvin).
P_W = Partial pressure of H_2O at ambient temperature in torr.
P_A = Ambient pressure in torr.

Note: *The* $\left(\left(1 - \frac{Pw}{Pa} \right) \right)$ *expression applies only if using a bubble flowmeter.*

EXAMPLE 2: Known Concentration of Sample in Detector

$$S = \frac{E}{Cd}$$

Where:

S = Sensitivity in mV x mL/mg
E = Detector signal (peak height) in mV
C_D = Concentration of test substance in the measured peak volume in mg/mL

Peak volume =
Peak width at half height (in min.) x gas flow rate at detector (in mL/min) = W • h/2 x FC

Calculate Detector Detectivity

To calculate the detectivity of the TCD, proceed as follows:

$$Detectivity = \frac{2 \times Noise}{Sensitivity}$$

Adjust TCD Carrier Gas Flow Rates

All 450-GCs equipped with TCDs are factory tested at the flow rate listed below.

Carrier Gas:	Helium
Inlet Pressure:	80 psig
Purity:	99.999%
Column Flow Rate:	30 mL/min
Reference Column Flow Rate:	30 mL/min

MAINTENANCE

The Thermal Conductivity Detector is not user serviceable and has no replacement parts. To condition the TCD, set the detector temperature to 350°C and allow it condition for 4 hours or overnight. The Electronics (filaments) box must be Unchecked (switched OFF) during this procedure.

TCD Test Chromatogram

For a TCD test chromatogram see section Detector Test Chromatograms, on page 422.

ELECTRON CAPTURE DETECTOR (ECD)

WARNING:
RADIATION SOURCE

The Electron Capture Detector contains a beta-emitting radioactive isotope, Ni^{63}. Users of this detector are required by regulations of the Nuclear Regulatory Commission (NRC) to read the radiation safety procedures described in the Radiation Safety Manual (03-913999-00). Manual is included with the ECD.

The electron capture detector (ECD) is a highly sensitive detector capable of detecting picogram amounts of specific types of compounds. The high selectivity of this detector can be a great advantage in certain applications. Compared with the FID, it has much more limited linear response. The response can vary significantly with temperature, pressure and flowrate.

The ECD is installed on the detector oven, directly above the column oven. The ECD electronic Board is installed in the electronics cabinet. The electronics of the ECD are controlled through the 450-GC keyboard, whereas gas flows are set and controlled at the pneumatics panel and in the Adjustments section of the Method if Detector EFC is installed.

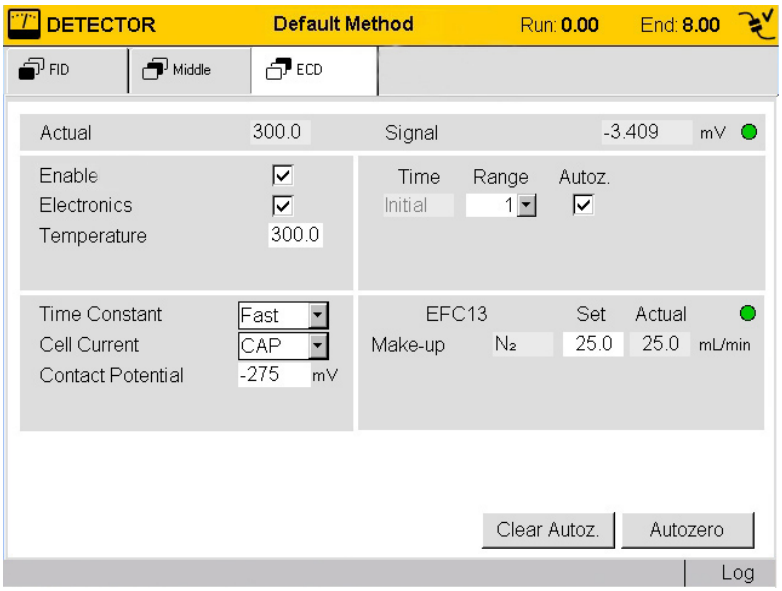
An ECD equipped with a detector cell is included in the ECD Kit. Varian is authorized to distribute this kit to General Licensees, as defined in the Radiation Safety Manual.
The Radiation Safety Manual(s) can be found on the CD-rom CP501425 which is included in the ECD package.
Due to the NRC regulations on the testing, packaging and labeling of radioactive materials, additional or replacement ECD detector cells are available only as kits.

Initial Set-up

To set up the ECD, proceed as follows. Note that when the 450-GC is first powered up all heated zones with the exception of the column oven are powered off and all detector electronics are turned off.

1. Refer to the Pre-Installation Instructions for additional information on gas supply requirements.

2. Press the DETECTOR key on the 450-GC Display/Keyboard to display the DETECTOR screen. The ECD should be listed in Position Front (), Middle () or Rear (). If your GC is equipped with detector EFC verify the makeup gas you will be using with your ECD.



3. Install a chromatographic column in the GC, on page 13. If the analytical column is not pre-conditioned, use a no-hole ferrule in the detector column nut while conditioning the column and leave the detector end of the column loose in the oven. If the analytical column is pre-conditioned, follow the usual procedure for column installation.

ECD Adjustments

There are ECD adjustments which should be set before operating the detector.

Cell-Current

In order to obtain the optimum linear and dynamic ranges of response, you must select the proper ECD cell current. Your choice of cell current depends on the specific carrier gas that is used and the baseline signal (frequency, f_0) with only carrier gas present. One of 4 values may be selected for the cell current:

- Ar/CH_4 (1710 pA)
- CAP (480 pA)
- N_2STD (290 pA)
- N_2HIGH (146 pA)

In addition, a Zero cell current setting is provided for convenience in setting the cell contact potential (see below).

For normal operation with nitrogen carrier gas, use the N_2STD setting. When a greater linear range is desired, select the Ar/CH_4 setting and use 90% Argon/10% Methane as the carrier gas.

Set the switch to CAP when using capillary columns. For capillary columns, the column bleed is low and the baseline is usually below 1 mV when current is set to N_2STD on Range 1.

You may encounter applications in which high column bleed is unavoidable when using nitrogen as the carrier gas. In such instances, the baseline signal may be too high. Select the N_2HIGH setting in these situations.

In general, the baseline signal should never exceed 25 mV when measured on Range 1. A value above 25 mV indicates that you have selected the wrong reference current for the chromatographic conditions (column, phase, sample matrix). A baseline signal above 25 mV decreases the useful linear range of the ECD detector.

Time Constant Switch

The time constant has 2 settings: SLOW (200 msec) and FAST (50 msec). For most applications, select the SLOW setting. For capillary applications with peak widths at half height of less than 2 seconds, select the FAST setting.

Setting the Contact Potential

An extremely clean ECD system can yield a contact potential that must be nulled for optimum performance. The contact potential may vary with time and detector temperature for several days after the cell is first put into operation, therefore you may need to repeat this adjustment procedure daily until the contact potential stabilizes.

Install a conditioned column and establish carrier gas and makeup flow. If the column is not well conditioned, remove the column from the detector inlet and cap off the inlet to the ECD. Condition the column as recommended by the supplier, then reconnect the column to the detector. **NEVER** operate the ECD cell at high temperature without a source of inert gas, either carrier gas from the column or makeup gas. Conditioning a column connected to the ECD detector can severely contaminate the detector, requiring a costly cleaning to restore proper function!

1. Set the ECD detector to the desired operating temperature for your application, typically 300 °C and setting the Oven Power to ON.
2. Turn the Electronics ON and set the Range to 1[a].
3. Un-check the AUTOZ. (Autozero) box.
4. Allow the detector to come to temperature and equilibrate for several hours, or preferably overnight.
5. Press the Clear Autoz. button.
6. Select a Cell Current of Zero.
7. Set the contact potential to -760 mV.
8. The ECD signal on the 450-GC display should read between -12.7 and -13.5 mV.
9. Increase the contact potential to +760 mV.
10. The ECD signal should increase by several mV, but may still be negative.
11. Now adjust the contact potential in a negative direction until the ECD signal is within 0.5 mV of its original value, e.g., if the signal was -12.7 mV, adjust the contact potential until the ECD signal reads -12.2 mV.
12. Now set the cell current to the desired value.

The Clear Autoz. button clears the previously stored detector background signal level.

[a] Range 10 is the most sensitive and preferred range for analytical use. If the range is set to 1, the signal will be attenuated with a factor 10, the signal value will be 10 times lower then range setting 10.

ECD Operation

Observe the following procedures when operating with an ECD.

Column and Septum Bleed

The ECD can become contaminated by chemicals that bleed from columns and septa. Such column and/or septum bleed can seriously decrease the ECD cell current which may result in an unacceptable increase in the output signal. To prevent such contamination, condition all columns and septa before you use them with the ECD.

Column Conditioning

NEVER condition a column while it is connected to the ECD detector. Instead, remove the column from the detector, cap the ECD column fitting with a nut and a no-hole ferrule. Maintain makeup gas flow to the detector to keep the ECD cell clean. Condition new columns for a minimum of 10 hours at 40 °C below the maximum temperature of the column stationary phase, or as directed by the column supplier. Complete the column conditioning phase before you connect the column to the ECD.

Column Oven and Detector Oven Temperature Selection

In selecting ECD oven temperatures, consider the following:

- The maximum permissible operating temperature of the radioactive foil is 400 °C. This foil temperature cannot be reached, even when the detector oven temperature is set to 450 °C. Do not add additional insulation material around the detector tower. Added insulation can raise the foil temperature and reduce its operating lifetime.
- A high detector oven temperature reduces the likelihood of contaminating the foil with material bleeding from the column.
- The higher the temperature of the foil, the greater the rate of migration of the ^{63}Ni into the backing material. Rapid migration of the ^{63}Ni reduces the electron flux and the lifetime of the foil.
- Background noise increases with an increase in the detector and column oven temperatures.

Use these guidelines to select the detector oven temperature:

- Detector temperature should be at least 30 °C higher than the maximum column oven temperature, but not lower than 150 °C.
- To prevent column bleed from fouling the detector, wait for the detector to reach its operating temperature before you increase the column temperature.

ECD Pneumatics

The 450-GC is tested at the factory at the flow rates listed in Table 10 on page 26. Use these flow rates as a starting point. Measure the flow rate at the exit tube of the ECD cell.

You may need to clean the detector cell periodically with carrier gas. Flow rates for the thermal cleaning procedure are also listed in Table 10 on page 26.

Refer to the installation section for a list of gas filters recommended for packed *column or capillary applications.*

Each ECD kit is equipped with a Teflon® flow tube assembly. When setting/checking the flow rate, attach the end of the flow tube with the ferrule to the 1/16-inch OD exit tube of the ECD cell. Attach the other end of the flow tube assembly to a flowmeter. To prevent cross-contamination between detectors, use the flow tube only with the ECD and store the assembly with the ECD kit.

WARNING:
RADIATION SOURCE

After prolonged use of the ECD detector, radioactive contaminants may build-up on the detector cap and exit tube. In such cases, it is possible that you could come in contact with radioactive material when checking or adjusting the carrier gas flow rate. Refer to Radiation Safety Manual.
Always wash your hands after handling the top of the ECD.

Gas	*Inlet Pressure at Gas Cylinder*	*Minimum Purity*	*Recommended Flow Rate (mL/min)*
Carrier: N_2, He, or Ar/CH_4 (90:10)	150 psig	99.999%	30 (packed column) ≤5 (capillary columns)
Detector Make-Up (capillary): N_2 or Ar/CH_4	150 psig	99.999%	20 to 30

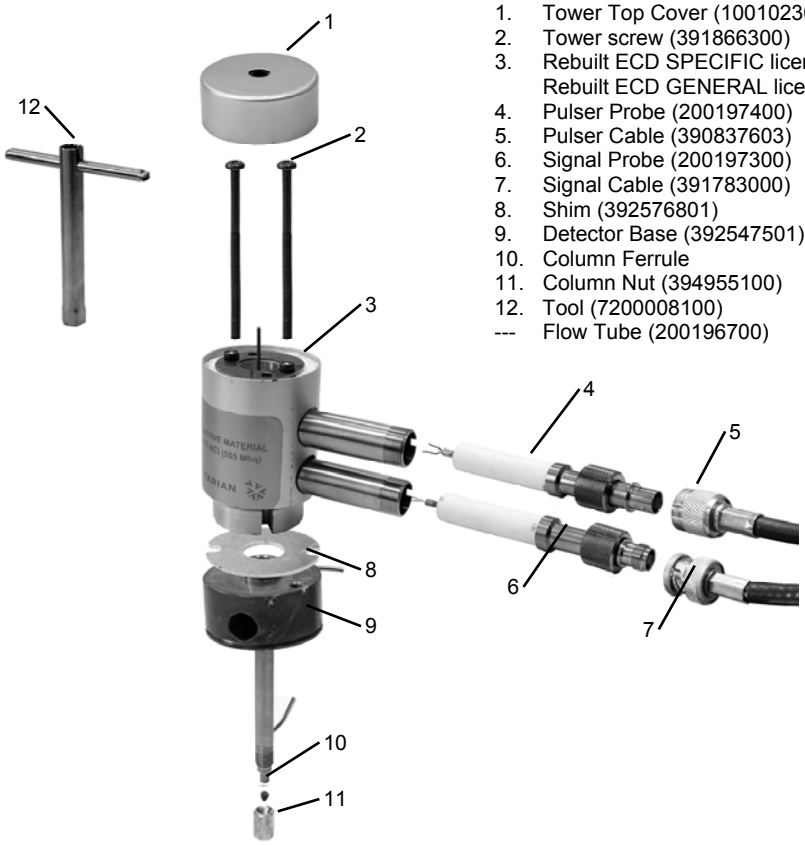
Table 52: ECD Gases, Pressures, Purity and Flow Rates

Installation/Disassembly

WARNING:
RADIATION SOURCE

In the ECD Kit (P/N 02-001972-00) the detector cell, which contains the radioactive ionization source, is fixed within the detector tower and cannot be removed. Do not attempt to remove the cell from the tower. Regulations for the handling of radioactive materials strictly prohibit unlicensed disassembly. Evidence of removal voids the warranty. Violations of these regulations are reported to NRC.

Exploded View



1. Tower Top Cover (100102300)
2. Tower screw (391866300)
3. Rebuilt ECD SPECIFIC license (200197271)
   Rebuilt ECD GENERAL license (USA only 200197270)
4. Pulser Probe (200197400)
5. Pulser Cable (390837603)
6. Signal Probe (200197300)
7. Signal Cable (391783000)
8. Shim (392576801)
9. Detector Base (392547501)
10. Column Ferrule
11. Column Nut (394955100)
12. Tool (7200008100)

--- Flow Tube (200196700)

Figure 56: ECD Exploded View

If you need to remove the detector components to inspect, clean, or replace parts, or to install another detector, refer to the following disassembly/re-assembly instructions for the ECD.

1. Cool the ECD to 50 °C.
2. Remove the detector tower cap and insulation.
3. Remove the signal probe and pulser probe.
4. Insert the installation tool into the detector tower assembly (located over the detector cell). Loosen the hex nut until the cell turns freely.
5. Remove the 2 tower mounting screws.
6. Remove the detector from the detector oven.

After removing the ECD, place the cap on the tower and place the cooled detector and installation tool in the ECD case. Store the detector in an area that is clean and of a low humidity. Refer to the Radiation Safety Manual for instructions on the storage and shipping of the ECD detector.

If the ECD is to be transferred to another GC, transfer the “CAUTION RADIOACTIVE MATERIAL” label to the front of the other GC as well. However, if you removed the detector only to gain access to the detector oven and you plan to reinstall the detector, place the ECD in a clean, uncontaminated area and proceed to the next paragraph.

ECD Installation

Because of the sensitivity of the ECD to oxygen, check the pneumatics for leaks prior to its installation.

1. Turn the detector oven power OFF.
2. Remove the Detector cover and the GC Top-Cover.
3. Remove both probes (pulser and signal) from the tower arms (see Figure 56, on page 335) of the detector assembly.

Be sure both probes have been removed from the detector tower arms before attempting to install the detector tower. If you insert the installation tool before you have removed the probes you will damage the probe electrode contacts.

WARNING

NON-SERVICEABLE ASSEMBLY. Do not attempt to remove the cell assembly which is fixed inside the tower.

4. Check to see whether the detector tower, base and detector cell fittings are clean. Install the ECD shim on the detector base.

Poor metal-to-metal contact between the tower contact area and the detector oven can produce a temperature gradient.

5. Remove the insulated detector cap.
6. Position the tower assembly on the detector base and install the 2 tower mounting screws loosely in the tower assembly.

The tower mounting screws must not be over tighten.

7. Insert the installation tool into the detector tower assembly (located over the detector cell). Engage the hex nut at the lower end of the cell assembly. Tighten the cell assembly into the base to provide a leak-free seal. Do not over-tighten.
8. Alternating between the 2 tower mounting screws, evenly tighten the tower into place.

9. Carefully install the signal and pulser probes in the tower arms. The signal probe (bottom) has a twist-lock cable connector and a narrow clip to engage the collector cylinder. The pulser probe (top) has a threaded cable connector and a wide clip to engage the foil cylinder. Insert the probes into the tower arms with the keys on the probes lined up with the notches in the tower arms. If you encounter any resistance during the insertion of the probes, check that the probes are being installed in the correct positions.

10. Install the insulated detector tower cap. The label shown is factory installed on a detector tower registered as a General License Device or Specific License.

Figure 57: Radioactive Caution Label – General License Device

Figure 58: Radioactive Caution Label – Specific License

11. Connect the pulser and signal cables to the appropriate probes of the ECD. Refer to the designation on each cable connector.

12. Cable connections for the operation of the ECD require installation of the ECD Electrometer Board.

13. Connect the gas supplies at the rear of the GC.

14. Replace the GC Top-Cover and the Detector cover.

15. Install the “CAUTION RADIOACTIVE MATERIAL” label (P/N 31-000347-00) in a location on the front of the GC that is clearly visible. This label describes the ECD radioactive isotope and the amount of radioactivity.

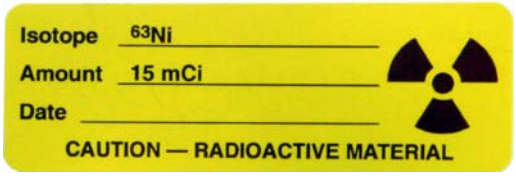


Figure 59: Radioactive Caution Label

16. Return the installation tool to the ECD case. You will use this tool each time you remove the ECD. Retain the ECD case should you need to store the ECD or to return the detector to Varian. See the Radiation Safety Manual.

MAINTENANCE

Chemical contaminants may deposit on the components of the detector assembly as the ECD is operated. In many cases, you can remove contaminants deposited on the radioactive foil or cell ceramic insulators by heating.

Refer to the Radiation Safety Manual (P/N 03-913999-00) for detector repair, radioactive decontamination, foil replacement and leak-test information.

Although all heated zones on the 450-GC have a potential upper temperature of 450 °C, Varian recommends that you do not operate the ECD above 420 °C. (The radioactive source temperature is less than 400 °C even when the detector set temperature is 450 °C).

Thermal Cleaning With Carrier Gas

Use N_2 or Ar/CH_4 as the make-up gas to thermally clean the ECD. Refer to Table 10, on page 26 for flow rates.

1. Remove column from the detector, cap the detector inlet using a no-hole ferrule (28-694590-01) and column nut (03-949551-00).

2. Confirm makeup gas is flowing out of the ECD vent tube, flow should be at least 30 mL/min. Increase the DETECTOR TEMP to 400 °C.

WARNING:
RADIATION SOURCE

Do not attempt to chemically clean the source. Submerging the cell in liquid or running a liquid through the cell will cause radioactivity to migrate, contaminate the exterior of the cell and damage the insulators. The cleaning of the radioactive ECD source can by performed only by an NRC licensee. Cleaning of the ECD source by persons other than registered NRC licensees is strictly forbidden by law.

CAUTION

AVOID FOIL DAMAGE. The carrier gas must be flowing through the detector cell before setting the DETECTOR TEMP above room temperature. Failure to do so may damage the radioactive foil, which is not covered by warranty.

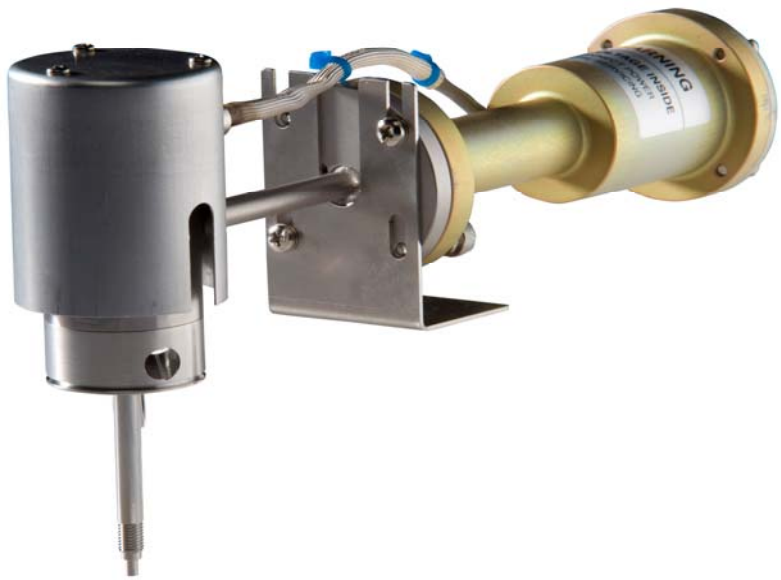
3. Monitor the output signal while the detector heats to its maximum temperature. The signal will increase initially, then gradually decrease as chemical contaminants vaporize from the detector surfaces.

4. Allow the DETECTOR TEMP to remain at 400°C until the signal reaches a stable level. A stable signal indicates that the foil has been cleaned as much as it can be by this method.

5. Set the detector at normal operating temperatures and flow carrier gas through the detector overnight. Check and readjust the Contact Potential setting if necessary.

6. If the performance of the detector is not restored by these cleaning methods, refer to the Radiation Safety Manual (P/N 03-913999-00) for information on replacement of the foil.

PULSED FLAME PHOTOMETRIC DETECTOR (PFPD)



Description

The Pulsed Flame Photometric Detector (PFPD) is the newest member of the family of flame-based gas chromatographic detectors. The PFPD operates in a pulsed-flame rather than continuous-flame mode and offers a number of significant improvements over the conventional flame photometric detector:

- Increased detectivity.
- Increased selectivity (relative to hydrocarbons).
- Decreased hydrogen and air consumption.

Basic Components of the PFPD

- PFPD assembly.
- PFPD electrometer board.
- PFPD pneumatics.

Features of the PFPD Assembly

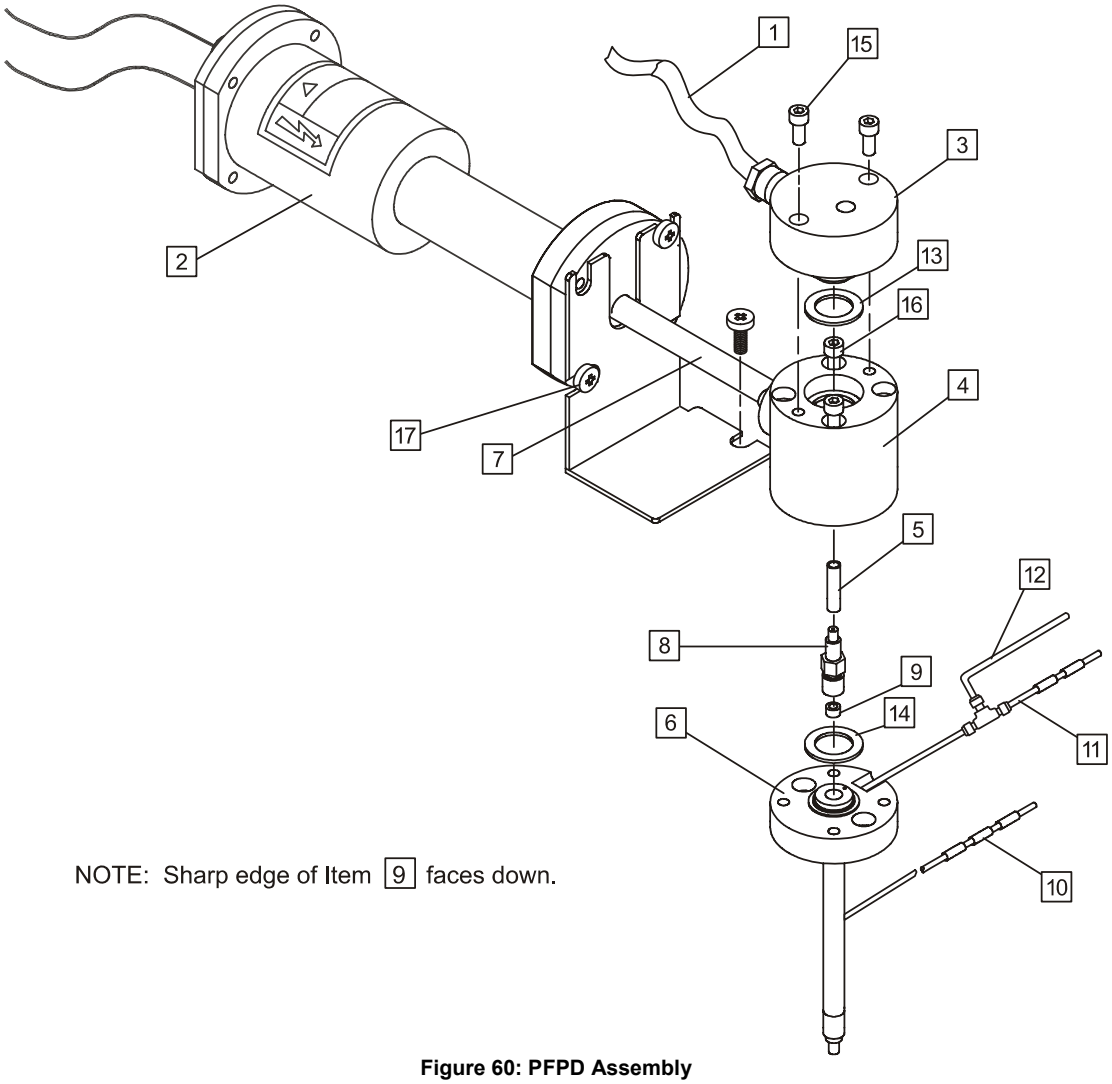


Figure 60: PFPD Assembly

Item	*Description*	*Function*
1	Igniter coil assembly	Provides the heat that ignites the flame.
2	Photomultiplier tube (PMT)	Converts the photons emitted in the flame into a current for measurement.
3	Igniter cap	Holds the igniter coil and minimizes reflected light from the igniter coil.
4	Detector tower	Supports the optics, quartz combustor tube and igniter cap.
5	Quartz combustor	A transparent inert surface that contains the column effluent for combustion and the transmission of emitted light.
6	Detector base	Holds the detector tower and secures the chromatographic column to the detector.
7	Light pipe	Carries light from the combustion region toward the optical filter and photomultiplier tube.
8	Combustor support	Holds and positions the quartz combustor within the detector tower.
9	Combustor support seal	Seals the space between the combustor support and detector base.
10	Hydrogen-air center flow inlet	Hydrogen-air mixture enters here and flows up through the center of the quartz combustor.
11	Hydrogen-air wall flow inlet	Hydrogen-air mixture enters here and flows along the outside wall of the quartz combustor and into the igniter chamber.
12	Air2 inlet	Air2 enters here and mixes with wall flow.
13	Crunch washer, upper	Seals the space between the igniter cap and detector tower.
14	Crunch washer, lower	Seals the space between the detector tower and detector base.
15	Allen screws, 9/64"	Secures the igniter cap to the detector tower.
16	Allen screws, 8/32 x 1-1/2"	Secures the detector tower to the detector base.
17	Phillips-head screws, 8/32 x 3/8"	Secures the photomultiplier tube assembly to the support bracket.

Features of the PFPD Electrometer Board

J3
IGNITOR

J2
PMT HV

J1
PMT SIGNAL

Description	Function
J1: Electrometer input signal cable BNC connector	Photomultiplier signal output is directed to the electrometer board through this BNC connector.
J2: Photomultiplier tube high voltage cable TNC connector	High voltage source on the electrometer board is directed to the photomultiplier tube through this TNC connector.
J3: Igniter cable Molex connector	Current to the igniter coil is directed through this Molex connector.
SIGNAL (TP1): Test point	With an oscilloscope, used to set the Gate Width and Gate Delay.
TRIGGER (TP2): Test point	Used to monitor the gate trigger (threshold).
GATE (TP3): Test point	Used to monitor when the integration period (Gate Width) begins.
J4: Top • Signal • Trigger • Gate • Ground	These are the same signals provided at TP1, TP2 and TP3, organized to provide easy connection to a cable. Used to connect to the PFPD Analytical Software.

Theory of Operation of the PFPD

By limiting the flow rates of air and hydrogen to approximately one tenth those typically used in an FPD, the PFPD achieves substantial improvements in both detectivity and selectivity. At these low flow rates, a continuous flame cannot exist.

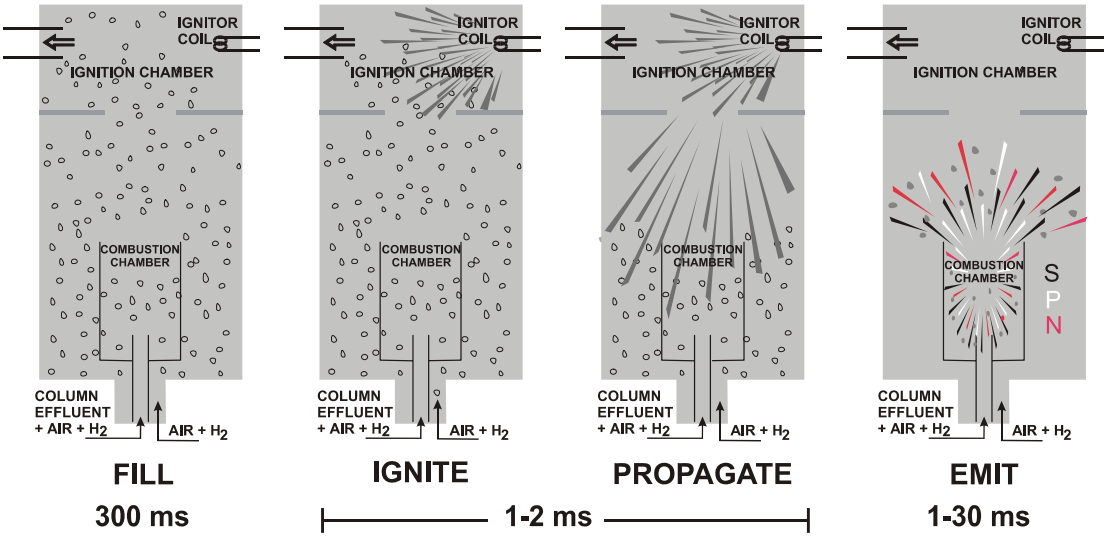
Instead, the combustor and igniter chambers fill with the combustible mixture of air and hydrogen. When this mixture enters the igniter, the mixture ignites and a flame front propagates back through the combustor, terminating when the flame reaches the combustor support.

The detector then refills with the combustible mixture and the process repeats.

Stages of Flame Pulsation

The flame in the PFPD pulses because the flow rates of hydrogen and air will not sustain continuous combustion. Flame pulsation involves 4 stages:

- **Fill**: Air and hydrogen mix and enter the combustion chamber at two points. Part of the combustible gas stream mixes with the column effluent and moves upward through the interior of the quartz combustor tube. A second part of the gas stream sweeps along the outside of the quartz combustor tube and into the igniter chamber.
- **Ignite**: The igniter chamber contains a continuously heated igniter coil. When the combustible gas mixture reaches the heated coil, the mixture ignites.
- **Propagate**: The flame front propagates downward into the combustion chamber. At the bottom of the combustion chamber, the flame extinguishes. During this propagation phase, the sample molecules in the flame are broken down into simpler molecules or atoms.
- **Emit:** During and after flame propagation, the sample atoms of interest undergo further reaction to form electronically excited species, from which light is emitted. The flame background emission is complete within less than 3-4 milliseconds after propagation, whereas phosphorus and sulfur molecular species emit over a much longer time. This difference in emission times yields the enhanced detectivity and selectivity of the PFPD.



How Does the PFPD Achieve its Greater Detectivity and Selectivity?

The enhanced detectivity and selectivity of the PFPD are a consequence of two main factors:

- Separation (in time) of the specific element emission from the background emission.
- Concentration (in time) of the emitted light.

Background Emission in a Pulsed-flame

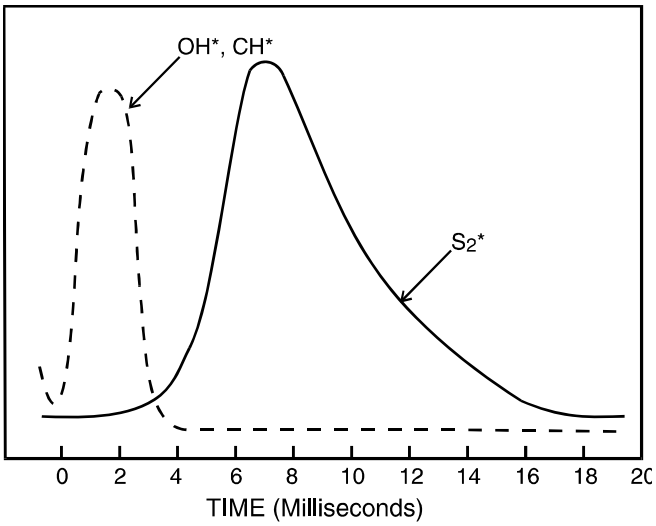
Like the conventional FPD, the PFPD utilizes an air:hydrogen flame. Hydrogen-rich flames favor a variety of gas phase chemical reactions, some of which yield molecular products that emit light (e.g., chemiluminescent reactions). Important excited combustion products include CH*, C_2* and OH*. The light from such combustion products is referred to as the *background emission*. Hydrogen-rich air:hydrogen flames typically yield chemiluminescence hydrocarbon background emission in the blue region with a maximum near 436 nm.3

Time-dependence of Emission Profiles

An important advantage of pulsed-flame operation is the time-dependence of the resulting emission profiles. Flame pulsation enables the molecular emissions of sulfur- and phosphorus-containing compounds to be separated or resolved in time from the flame background emission itself. This resolution is possible because the emission time of the flame background is much shorter than the emission time of the excited molecular products of compounds of analytical interest. As an example, CH* and OH* emission in a hydrogen-rich flame occurs within the time it takes the flame to propagate through the combustor, typically 2 to 4 msec. In contrast, S_2* emission reaches its maximum 5-6 msec after the CH* and OH* emission has ceased. This separation in time enables the sulfur emission to be monitored when the flame background has dropped to a negligible level. The result is a great improvement in the detectivity and selectivity of sulfur and phosphorus detection.

Emission Profiles

The following figure shows the hydrocarbon and sulfur emission profiles as a function of time (Cheskis, S.; Atar, E.; Amirav, A. *Anal. Chem.* **1993**, 65, 539).



Concentration (in time) of the Emitted Light

In the PFPD, detectivity is greater because all the emission occurs during and immediately following the flame pulse. This increases the brightness of the analytical signal relative to the dark current and other electronic noise. For example, at a typical pulse rate of 2.5 Hz, all the sample eluting from the column during 400 msec is burned in a single pulse, emitting over perhaps 10 msec. This results in a signal which is not only approximately 40 times as bright as that from a continuous flame source, but also is resolved in time from the interference of the flame background.
In addition to the general improvement in detectivity and selectivity as a result of flame pulsation, a further increase in the relative emissions of elements of interest is achieved through the appropriate choice of gas composition. As an example, the formation of S_2* is favored in cooler hydrogen-rich air:hydrogen flames. Hydrogen content has less effect on phosphorus emission.

Separation of the Element Emission from Background Emission

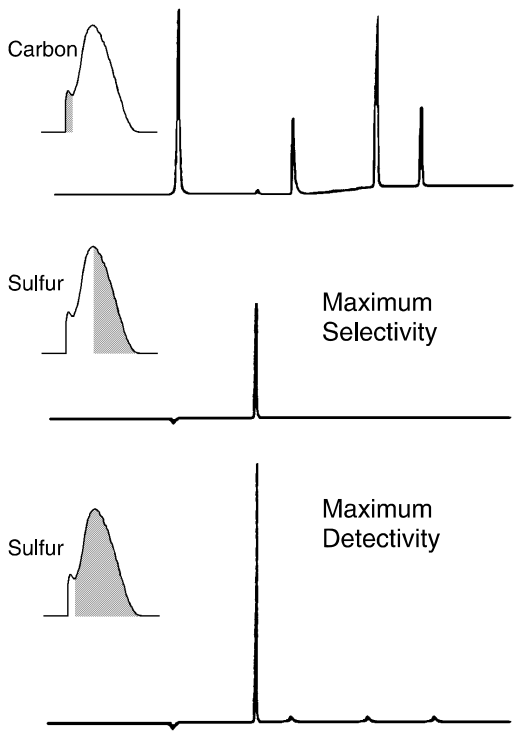
The flame pulsation in the PFPD yields *time* as an added dimension to flame photometric detection. Both selectivity and detectivity are improved through the ability to:

- Resolve emissions of sulfur or phosphorus from the flame emission background.
- Resolve emissions of sulfur and phosphorus from each other.

As mentioned, the emission times in an air:hydrogen flame of such excited molecular entities as S_2* or HPO* are generally much longer than the emission times of species responsible for the background emission (e.g., CH*, C_2* and OH*). This delay allows the sulfur or phosphorus emission to be integrated after the flame background has dropped to a negligible level. The result is a lower overall noise level and thereby greater detectivity.
The electrometer Gate Delay and Gate Width are used to select the time period of the emission that is to be integrated. In this way, only a small segment of the background noise is integrated during any pulsation. For example, if the time between pulses is 400 msec and the Gate Width is set to 20 msec, 95% of the total background noise is discarded, which further contributes to an improvement in detectivity.

Effect of Gate Control

The following figure illustrates the effect of Gate Delay on selectivity and detectivity.

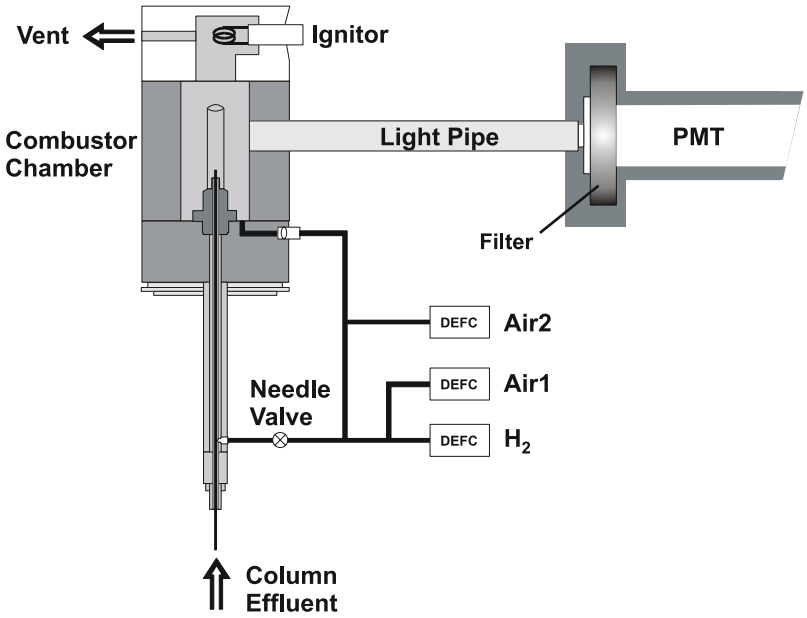


Adjustment of the Gas Composition

The flow rates of air and hydrogen affect the detectivity and selectivity of the PFPD. The total flow rates of air and hydrogen set the operating frequency (rate of pulsation) of the PFPD. Typical operating frequency for the PFPD is near 2-4 pulses/second. The relative proportions of air and hydrogen influence the selectivity by favoring the formation of an emitting species or suppressing the formation of an unwanted interfering species. For example, when detecting phosphorus pesticides in plant tissues, the use of a slightly more air rich flame than usual will suppress formation of the interfering S_2^* molecule, improving P/S selectivity.

PFPD Flow Path

The figure below shows the flow path of gases entering and exiting the PFPD.



The air:hydrogen mixture enters the PFPD at two points: around the outer wall and through the center of the quartz combustor tube. The wall flow is used to refill the upper igniter chamber of the detector after each pulse, while the combustor flow mixes with the column effluent and refills the combustor. For proper operation, the combustor should refill just slightly faster than the igniter. The refill rates are balanced by use of a needle valve on the air:hydrogen mixture and by the flow of Air2, which bypasses the combustor and goes directly to the igniter.

References

Refer to the following articles for additional information on the PFPD.

- S. Cheskis, E. Atar and A. Amirav, "Pulsed Flame Photometer - A novel Gas Chromatography Detector," Anal. Chem., 65 539-555 (1993).
- Amirav and H. Jing, "Pulsed Flame Photometer Detector for Gas Chromatography," Anal. Chem., 67, 3305-3318 (1995).
- H. Jing and A. Amirav, "Pesticide Analysis with the PFPD and a Novel Direct Sample Introduction Device," Anal. Chem., 69, 1426-1435 (1997).
- H. Jing and A. Amirav, "Pulsed Flame Photometric Detector, A Step Forward towards Universal Heteroatom Selective Detection," J. Chromatog. A., 805, 177-215 (1998).
- J. A. Jacobsen, F. Stuer-Lauridsen and G. Pritzl, Organotin Speciation in Environmental Samples by Capillary Gas Chromatography and Pulsed Flame Photometric Detection (PFPD) Appl. Organometallic. Chem. 11, 737-741 (1997).
- Amirav and H. Jing, "Simultaneous Pulsed Flame Photometric and Mass Spectrometric Detection for Enhanced Pesticides Analysis Capabilities," J. Chromatog. A., 814, 133-150 (1998).

Specifications

Specification	***Sulfur Performance***	***Phosphorus Performance***
Detectivity	1 pg S/sec	0.1 pg P/sec
Selectivity	$\geq 10^6$ S/Carbon **NOTE:** The selectivity of Sulfur/Carbon is a function of the sulfur concentration at any time (from 10^5 near its detection limit to $\geq 10^6$ at higher sulfur concentrations).	$\geq 10^6$ P/Carbon
Linear Dynamic Range	10^3	10^5
Drift (in 20 minutes)	10 x peak to peak noise	10 x peak to peak noise

Table 53: Specifications PFPD

Nitrogen or Other Element Operation

The nitrogen mode enhancement to the Pulsed Flame Photometric Detector extends the range of wavelengths available for detection of element-specific emissions to 900 nm in the near infrared. This not only allows detection of the nitrogen emissions between 700 nm and 800 nm, but also permits the detection of other elements, such as tin and arsenic. Section 4 of this manual gives details of operation in the nitrogen mode, as well as some preliminary information on detection of additional elements.

Nitrogen Mode Specifications	
Detectivity:	20 pg N/sec
Selectivity:	$>10^5$ N/C
Linear Dynamic Range:	$>10^2$
Drift:	<10X peak to peak noise in 20 minutes

Table 54: Specifications Nitrogen Mode PFPD

Installation

Inspecting the PFPD

The PFPD is installed and tested at the factory before the GC is shipped.

Carefully inspect the PFPD. If there is evidence of damage or if parts are missing from the PFPD or its accessory kit, please contact your nearest Varian, Inc. subsidiary or Varian, Inc. representative.

PFPD Accessory Kit

The PFPD accessory kit (03-925190-90) contains tools (Items 1-9 below) to disassemble the PFPD and a supply of parts used in its routine maintenance.

Item	Part Number	Qty	Description
1	03-925180-00	1	Teflon combustor extraction device (wide bore, 3 mm ID)
2	03-925181-00	1	Teflon combustor extraction device (narrow bore, 2 mm ID)
3	03-925192-00	1	Combustor support wrench
4	29-898500-00	1	Teflon tweezers
5	03-925194-04	1	Allen wrench, 9/64" (cleaned)
6	15-003347-00	15	Crunch washers (Aluminum)
7	03-925138-00	10	Combustor support seal (Aluminum)
8	88-189610-00	1	Teflon pipe thread tape, not shown
9	16-000327-00	1	Reducing union (flow check)

Table 55: Accessory Kit PFPD

Please note that Item 16, Sulfur Linearization Software, is not used with the 450-GC PFPD. Several important parts for Sulfur mode include Items 10, 11 and 12 while parts for Phosphorus mode include Items 13, 14 and 15.

Item	Part Number	Qty	Description
10	03-925176-00	1	Quartz combustor (2 mm ID, Sulfur)
11	03-925178-00	1	Combustor support (2 mm ID, Sulfur)
12	03-925151-01	1	Filter assembly, Sulfur
13	03-925177-00	1	Quartz combustor (3 mm ID, Phosphorus)
14	03-925179-00	1	Combustor support (3 mm ID, Phosphorus)
15	03-925151-02	1	Filter assembly, Phosphorus
16	-	1	Software, sulfur linearization software

PFPD Diffusion Source Kit

The PFPD diffusion source kit (03-925157-00) is supplied as an option to the PFPD.

Item	Part Number	Qty	Description
1	03-925132-00	1	Adapter seal
2	03-925195-00	1	Permeation tube, Sulfur
3	03-925196-00	1	Diffusion source holder
4	03-949786-00	1	Spring
5	03-925200-00	1	Injector adapter Cap
6	88-189610-00	1	Teflon pipe thread tape, 1/2" (not shown)

Table 56: Diffusion Source Kit PFPD

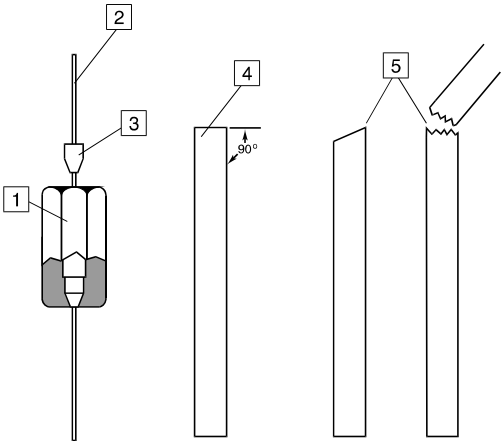
Installing a Chromatographic Column

The following procedure describes the installation (or reinstallation) of a gas chromatographic column in a Varian GC equipped with a PFPD.

To ensure good chromatographic performance with the PFPD, the tip of the column must extend 0.5-1.5 mm above the top of the combustor support.

Prepare the Column End

Item	Description
1	Capillary Column Nut
2	Capillary Column
3	Ferrule
4	Correct Column Cut
5	Incorrect Column Cut



Tools and Equipment Needed

These tools are needed to complete the following procedure.

Refer parts and supplies, on page 412.

- Tungsten ceramic scorer or a scoring tool.
- Magnifying lens.

Follow these steps to prepare the end of the chromatographic column for insertion into the PFPD.

1. Before cutting the end of the column, slide the capillary column nut (Item 1) over the fused silica column end (Item 2).

2. Install the appropriate Vespel®/Graphite or graphite ferrule (Item 3) over the column end.

Make certain that the tapered edge of the ferrule is toward the inside of the column nut (or away from the column end).

Typically, graphite ferrules contain sulfur as a contaminant. If you use graphite ferrules, expect a higher background during pre-conditioning of the chromatographic column. The background may be particularly high when using a new graphite ferrule that has not been used and conditioned in the past.

3. Hold the column securely between the thumb and forefinger of one hand. Use the scoring tool to score the column once lightly about 1 to 2 cm from the end of the column.

4. With your thumbs and forefingers, grasp the column on each side of the score and bend the column carefully away from the score mark until it breaks at the score mark.

5. Examine the cut with the magnifying lens to decide whether the column was properly cut (Item 4) or improperly cut (Item 5).

   If you observe small splinters of silica or bits of the outer coating on the column end, repeat the above procedure and make a fresh cut.

   If you observe a clean cut, you may use either of 2 techniques to install the column. If you wish to visually confirm the position of the column end above the combustor support, go to *Disassemble the PFPD, below*. If you would rather not disassemble the detector, see Page 360, *Insert the Column*.

Disassemble the PFPD

Refer to on page 344.

These tools are needed to complete the following procedure:

- Phillips-head screwdriver.
- 9/64" Allen wrench.
- Metal forceps.
- Teflon combustor extraction device.
- Teflon tweezers.
- Lab tissues.

1. Turn the column oven OFF to stop the oven fan.
2. Open the column oven door and wait for the column to cool to room temperature (or to a temperature at which you can comfortably handle the column).
3. Turn OFF the PFPD electronics and oven power and allow detector to cool to near room temperature.
4. Remove the Detector cover from the top of the GC.
5. Before using, thoroughly clean the 9/64" Allen wrench, 1/4" wrench, Teflon combustor extraction device and Teflon tweezer with methanol or iso-propanol. Set these tools aside on a clean lab tissue.

NOTE: Dust and dirt inside the combustor can interfere with emission and thereby lower the sensitivity of the PFPD. Therefore, never touch the internal parts of the PFPD with bare hands. Always use a clean lab tissue, Teflon combustor extraction device, or Teflon tweezer to remove or replace detector parts. Place parts on a clean lab tissue as they are removed from the PFPD.

6. Use the 9/64" Allen wrench to remove the 2 Allen screws (Item 15) from the top of the igniter cap (Item 3). Place the Allen screws on a clean lab tissue.

WARNING: BURN HAZARD ***The igniter cap and Allen screws may be very hot. Wait until the igniter cap and Allen screws have cooled or use metal forceps to remove the Allen screws from the igniter cap.***

7. Use the metal forceps to lift the igniter cap (item 3) from the detector tower (Item 4). **Place the igniter cap bottom up** on a clean surface (e.g., a clean lab tissue). The top of the quartz combustor (Item 5) is visible in the center of the detector tower.

WARNING: BURN HAZARD ***The igniter cap may be very hot. Wait until the igniter cap has cooled or use metal forceps to remove the igniter cap from the detector body.***

8. Gently insert the appropriate Teflon combustor extraction device into the opening in the top of the combustor. Wait 2-3 seconds for the Teflon combustor extraction device to seat, then lift the quartz combustor from the combustor support (Item 8). Place the quartz combustor on a clean surface (e.g., a clean lab tissue).
9. Use the metal forceps to remove the top aluminum crunch washer (Item 13) from the detector tower. Discard this used crunch washer.

Remove the Detector Tower (Optional)

If you wish to view the column insertion depth directly, or you wish to replace the combustor support, proceed with the following steps. If not, proceed on page 360.

1. Remove from the top of the detector tower the 2 Allen screws that hold the detector tower to the detector base. Also, remove the screw that holds the PMT assembly support bracket to the GC.

2. Hold the PMT assembly with one hand and the detector tower with the other. Carefully lift the detector tower straight up from the detector base. Set the detector tower with the PMT assembly aside on a clean surface.

3. Use the metal forceps to remove the bottom aluminum crunch washer. Discard this used crunch washer.

4. If you are changing combustor size, remove the combustor support using the clean combustor support wrench. Use the **clean** combustor support seal extraction tool (or clean capillary pick) to remove the combustor support seal. Replace with a new combustor support seal. Replace the previous combustor support with the new combustor support. Snug down the new combustor support with the support wrench but DO NOT OVERTIGHTEN.

5. If you are visually checking the insertion depth of the column, proceed to insert the column on page 360, ignoring the instructions regarding use of the Column Depth Gauge.

Insert the Column

Refer for details on page 344.

These tools are needed to complete the following procedure
Refer to parts and supplies, on page 412.

- Phillips-head screwdriver.
- 5/16" wrench.
- Capillary pick or combustor support seal extraction tool.
- Ceramic scoring tool.
- Magnifying lens.
- 3 mm combustor extraction tool/Column Depth Gauge.

To Insert the Column Visually

1. Check that the capillary column nut and the ferrule (reversed) are installed on the column as described in the manual. Check that approximately 20 cm of the detector end of the column is free so that it can be inserted into the detector. If you are reinstalling a used column, be sure that the burnt end of the column is trimmed off, leaving a clean, square end.

2. Insert the column into the detector and push the column up until it is approximately 1 mm above the end of the combustor support. Tighten the column nut to secure column in place. Next, proceed on page 361.

To Insert the Column Using the Column Depth Gauge

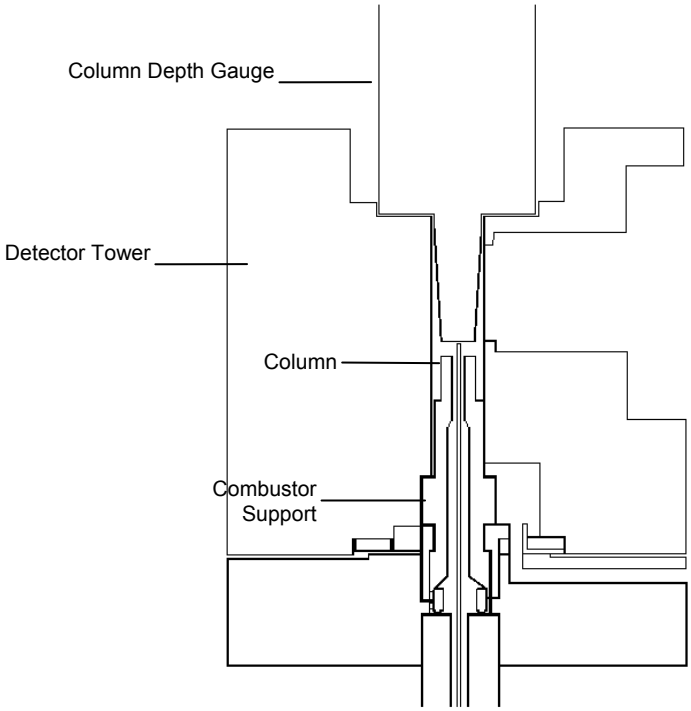
To insert the column with Column Depth Gauge on page 361 and follow instructions through step 3, then proceed as follows:

1. Check that the capillary column nut and the ferrule (reversed) are installed on the column as described in the manual. Check that approximately 20 cm of the detector end of the column is free so that it can be inserted into the detector. If you are reinstalling a used column, be sure that the burnt end of the column is trimmed off, leaving a clean, square end.

2. Insert the Column Depth Gauge into the hole from which the combustor was removed. Insert the column into the detector and push the column up until it begins to push up the depth gauge. Tighten the column nut gently until a small amount of force is required to move the column. Gently apply force to the depth gauge to push the column down until the depth gauge

contacts the detector tower. See the following figure. Tighten the column nut to secure the column in this position.

3. Continue with step 4 in Reassemble the PFPD.



Reassemble the PFPD

Refer on page 344.

Tools and equipment needed to complete the following procedure:

- Phillips-head screwdriver.
- 9/64" Allen wrench.
- Teflon combustor extraction device.
- Metal forceps.

1. Use the clean metal forceps to place a new bottom aluminum crunch washer (Item 14) on the detector base.

2. Hold the PMT assembly (Item 2) with one hand and the detector tower with the other. Sight the top of the combustor support (Item 8) through the detector tower. Keep the combustor support centered as you lower the detector tower (Item 4) over the support.

CAUTION

Be very careful not to crush the end of the column.

3. Reinstall the 2 Allen screws (Item 16) that hold the detector tower to the detector base (Item 6). Tighten the screws alternately. *Note: If installing the column with the aid of the column depth gauge, follow instructions* on page 360.

4. Use the Teflon combustor extraction device (of the correct taper) to insert the quartz combustor (Item 5).

5. Use the clean metal forceps to place a new top aluminum crunch washer (Item 13) on the detector tower.

6. Replace the igniter cap (Item 3) on the detector tower. Use the 9/64" Allen wrench to reinstall in the top of the igniter cap the 2 Allen screws (Item 15) that secure the igniter cap to the detector tower.

7. Reinstall the Detector cover on the top of the GC.

8. Turn on the PFPD electronics and PFPD Oven Power. Be sure that the detector gases are turned on. The detector may not begin pulsing normally until the detector tower has begun to heat up somewhat. Allow the detector to come to temperature and equilibrate for at least 15 minutes. During this equilibration period the polyimide coating on the portion of the column exposed to the flame is burned off, leaving a clean fused silica surface.

9. Did you install a quartz combustor with a different diameter?
   - If **Yes**, go to ***Optimizing the PFPD*** for instructions on optimizing the performance of the PFPD with the quartz combustor just installed.
   - If **No**, the PFPD is ready for operation.

Operation

Adjusting the Gas Flow Rates

The following procedure describes the adjustment of the flow rates of the gases to the PFPD. If you frequently adjust the column flow rate or change the carrier gas, you may want to calibrate the air and hydrogen flow controllers. Once the flow controllers are calibrated, you can quickly select new flow rates and use the optimization tables that are provided.

If the PFPD Detector EFC option is installed on your 450-GC, detector gas flows are set by pressing the Detector key, selecting the PFPD tab.
The Split Valve adjustment is described below.

Split Valve

A split valve is used to finely adjust the flow rate through the combustor section of the PFPD combustor. Follow these directions when adjusting this split valve:

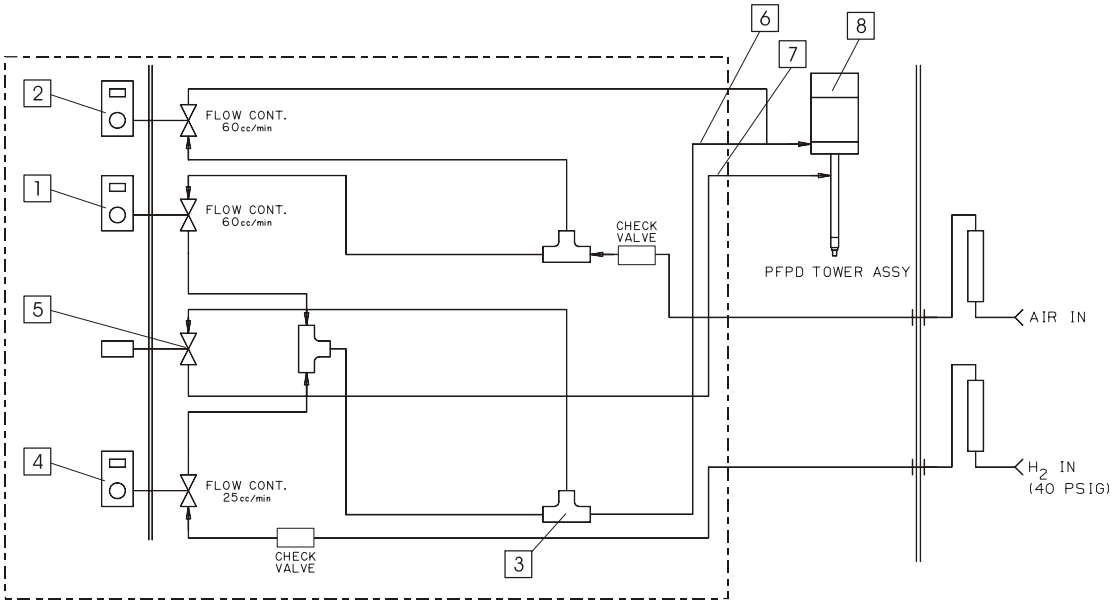
1. Disconnect the outlet line from the needle valve and replace with a flowmeter.

2. With Air1 and Hydrogen turned on, close the needle valve by turning the knob fully clockwise. The flowmeter should indicate a flow of less than 1 mL/minute. If this is the case, disconnect the flowmeter and reattach the outlet line.

3. If the flow from the needle valve exceeds 1 mL/minute, remove the knob by loosening the set screw. Rotate the valve stem clockwise gently until the flow from the outlet drops below 1 mL/minute. Do not overtighten the valve stem, as the needle valve does not need to shut off completely to function properly and overtightening can damage the delicate needle in the valve. After making the adjustment, reinstall the knob on the valve stem and secure with the set screw.

PFPD Gas Flow Diagram with EFC15

Refer to the following figure when adjusting the flow rates of the gases to the PFPD.



Item	*Description*
1	Air1 mass flow controller
2	Air2 mass flow controller
3	Center flow tee
4	Hydrogen mass flow controller
5	Split valve
6	Wall flow (H_2 + Air1 + Air2)
7	Center flow (H_2 + Air1)
8	Igniter cap

Adjust the Gas Flow Rates

Tools and equipment needed to complete the following procedure.
Refer to parts and supplies, on page 412.

- Varian Intelligent Digital Flowmeter **or** Soap flowmeter
- Stopwatch
- Flow measuring adapter (1/8"-to-1/16" Swagelok® reducing union)
- 1/4" flat blade screwdriver

For best stability of flows, flow controllers should have pressure applied to them for at least 24 hours before adjustments are made. A moderate flow of gas through the flow controller during this conditioning period is also helpful in stabilizing the flow controller.

1. Turn the igniter OFF.
2. Locate the *Air1* mass flow controller valve (Item 1). Note the current dial setting. Turn *Air1* OFF.
3. Locate the *Air2* mass flow controller valve (Item 2). Note the current dial setting. Turn *Air2* OFF.
4. Locate the *Hydrogen* mass flow controller valve (Item 4). Note the current dial setting. Turn *Hydrogen* OFF.
5. Install the flow measuring adapter (Swagelok 1/8" to 1/16" reducing union) to the threaded hole in the igniter cap. Connect a 2-3" piece of tube to 1/8" end of the union. Use Teflon thread tape to make the connection to the igniter cap gas tight. Measure and record the column flow rate, then turn the column flow OFF.

A Varian Intelligent Digital Flowmeter is highly recommended for this procedure.

CAUTION

Be sure the igniter seal is completely leak tight before attempting to measure flow rates. This seal can loosen due to temperature cycling during normal operation. This does not affect detector performance, but can lead to serious errors in setting flow rates. Tighten securely before beginning to measure flows.

6. Adjust the *Air1* mass flow controller valve until flow rate is near 17 mL/min. Write down and save the dial setting for the *Air1* mass flow controller valve. Turn the *Air1* flow controller Off.

7. Adjust the *Air2* mass flow controller valve until flow rate is near 10 mL/min. Write down and save the dial setting for the *Air2* mass flow controller valve. Turn the *Air2* flow controller Off.

8. Adjust the *Hydrogen* mass flow controller valve until flow rate is near 12 mL/min.

If you will be using hydrogen as your carrier gas, the total hydrogen flow to the detector should be approximately 12 mL/minute. The flow rate set in this step should thus be 12 mL/min minus the anticipated column flow rate. Write down and save the dial setting for the Hydrogen mass flow controller valve.

9. Restore the *Air1* and *Air2* flow controllers to the settings recorded above. Restore the carrier gas flow to the column. Remove the flow measurement adapter, remove any Teflon thread tape residue from the igniter cap.

Leave the flow controllers ON at all times unless you must turn them off to re-plumb or reconfigure the gas chromatograph. If you turn the flow controllers off for an extended period then on again, they must equilibrate overnight before the PFPD can achieve optimum performance.

10. Turn the PFPD and thereby the igniter ON.

Calibrating the Flow Controllers

For best stability of flows, flow controllers should have pressure applied to them for at least 24 hours before adjustments are made. A moderate flow of gas through the flow controller during this conditioning period is also helpful in stabilizing the flow controller.

Tools and equipment needed to complete the following procedure. Refer to parts and supplies, on page 412.

- Varian Intelligent Digital Flowmeter **or** Soap flowmeter.
- Stopwatch.
- Flow measuring adapter (1/8"-to-1/16" Swagelok® reducing union).
- 1/4" flat blade screwdriver.

1. Turn off the column flow. Or, if you want flow to continue to the column, disconnect the column from the detector and use a column nut and 1/16" no-hole ferrule to seal the detector.

2. Write down the current dial settings for Air1, Air2 and Hydrogen, then turn off each flow controller.

3. Install the flow measuring adapter (1/8" to 1/16" reducing union). Use Teflon thread tape to make a gas-tight seal.

A Varian Intelligent Digital Flowmeter is highly recommended for this procedure.

CAUTION

Be sure the igniter seal is completely leak tight before attempting to measure flow rates. This seal can loosen due to temperature cycling during normal operation. This does not affect detector performance, but can lead to serious errors in setting flow rates. Tighten securely before beginning to measure flows.

4. Connect the flowmeter to the adapter (Swagelok 1/8" to 1/16" reducing union). Turn the Air1 flow controller (clockwise) until the flowmeter indicates flow through the system. Slowly turn the Air1 flow controller (counter-clockwise) until the flow just stops. Again, write down the dial setting.

It is recommended that the flow controller dial be set so that it reads zero at this setting. Remove the knob then loosen the 2 set screws holding the dial to the flow controller shaft. While holding the flow controller shaft so that it does not rotate, turn the dial to 000. Retighten the set screws and replace the knob.

5. Set the dial to 100, 200, etc. and allow the flow to equilibrate. Measure the flow and record. When you have completed all measurements, turn off flow.

6. Repeat steps 1-5 for the Air2 and Hydrogen flow controllers.

Chart the flow rate versus dial setting so that you have a reference when setting flow rates as suggested in Optimizing the PFPD.

7. If you removed the column prior to calibrating the flow controllers, reinstall it now.

Optimizing the PFPD

The PFPD is optimized to maximize:

- Detectivity of Sulfur or Phosphorus.
- Selectivity of Sulfur and Phosphorus to Carbon or each other.
- Analytical throughput (shorten total analysis time).

Detectivity

Detectivity is the minimum detectable quantity (MDQ) of Sulfur or Phosphorus (usually expressed as picograms of Sulfur (or Phosphorus) per second. Detectivity is the preferred method of expressing the sensitivity of a detection method because it includes a consideration of the Signal-to-Noise ratio. Sensitivity alone is insufficient in that the sensitivity of the PFPD can be increased simply by increasing the voltage of the photomultiplier tube, but the noise increases as the signal increases, yielding little enhancement in the minimum amount detectable.

Selectivity

An important question when optimizing the selectivity of the PFPD is, "Selective relative to what?" In some situations, the Sulfur or Phosphorus response is best optimized relative to Carbon (the principal background emission in the air-hydrogen flame). At other times, it may be more important to optimize the Phosphorus response relative to the Sulfur response, as when Phosphorus is analyzed in certain plant tissue extracts that are high in Sulfur content.

Throughput

The additional selectivity of the PFPD can result in shorter analysis times and thereby greater sample throughput. As an example, by optimizing the sulfur response relative to hydrocarbons, the PFPD may detect fewer peaks thereby simplifying the chromatogram and increasing the chromatographic resolution. With greater resolution, the chromatography can be adjusted such that the peaks of interest elute faster thereby decreasing the analysis time. In addition, when hydrogen is used as the carrier gas, the PFPD can be operated at higher flow rates without greatly affecting the detectivity of the method. Higher carrier gas flow rates translates into shorter analysis times and greater sample throughput.

Suggested Fuel Gas Flow Rates

The following table lists flow rates for several carrier gases that are often used in common analysis situations when operating the PFPD. Use these flow rates as starting points when optimizing the performance of the PFPD.

Carrier Gas Flow Rate (mL/min)	*H_2 (mL/min)*	*Air1 (mL/min)*	*Air2 (mL/min)*
Helium			
2	13	17	10
5	13	20	10
10	20	34	10
Hydrogen			
2	11	17	10
5	9	18	10
10	15	31	10
Nitrogen			
2	14	17	10
5	16	21	10
10	24	34	10

Table 57: Flow Rates PFPD

You May Not Need to Adjust Flow Rates

The difference in optimum gas flow rates between sulfur and phosphorus is small. If you don't require the maximum sensitivity for an analysis, leave the flow rates as they are and change only the optical filter, gate delay and integration time, when shifting between the sulfur and phosphorus detection modes.

Suggested PFPD Adjustment Settings

The following adjustment settings are recommended for setup and routine operation of the PFPD. These adjustments are accessed by pressing the Adjustments button in the PFPD detector section of the 450-GC method.

Element	*Gate Delay*	*Gate Width*	*Trigger*
Sulfur	6	20	200
Phosphorus	4	10	200
Nitrogen	3.5	8	200

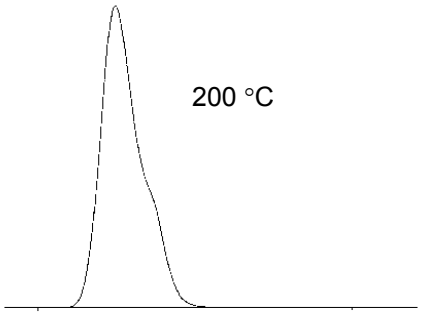
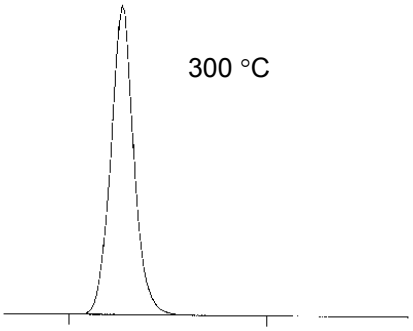
Optimization Criteria

Refer to the following table when optimizing the PFPD.

To Maximize ...	*Consider ...*
Phosphorus response	• Install wide bore quartz combustor. • Use moderately hydrogen-rich flame. • Adjust split valve such that detector is just out of tick-tock. • Set detector temperature ≥300 °C.
Sulfur response	• Install narrow bore quartz combustor. • Set hydrogen carrier gas flow rate to a value that is near, but just above, the minimum of the van Deemter curve for the column and maximum column temperature used. • Adjust split valve such that detector is just out of tick-tock. • Use optimum hydrogen-rich flame. • Increase Air2 to enhance ignition and flame propagation in the hydrogen-rich flame. • Set detector temperature ~200 °C. **NOTE: For some compounds, sulfur response decreases with increasing detector temperature. Set the detector temperature as low as possible while preserving chromatographic peak shape.**
Phosphorus-to-Carbon selectivity	• Install wide bore quartz combustor. • Use hydrogen as carrier gas. • Use *moderately* hydrogen-rich flame. • Adjust split valve such that detector is just out of tick-tock, then turn counterclockwise an additional 1/2 turn. • Detector temperature ≥300 °C. **NOTE: The Gate Delay can be adjusted to increase or decrease selectivity. However, as the selectivity increases, the detectivity often decreases. Set the Gate Delay to balance these performance criteria at levels that are appropriate to the application. Also, the gate should be set to open (or trigger) as close to the main peak as possible rather than before or after. Setting the gate correctly prevents the hydrocarbon and phosphorus responses from overlapping as the flame propagation slows in hydrocarbon-rich flames.**

To Maximize ...	***Consider ...***
Sulfur-to-Carbon selectivity	• Install wide bore quartz combustor. • Use hydrogen as carrier gas. • Use *moderately* hydrogen-rich flame. • Adjust split valve such that detector is just out of tick-tock, then adjust counterclockwise an additional 1/2 turn. • Set detector temperature ~200 °C. • Increase sample amount injected (inject larger volume or more concentrated form of sample). The trigger level should be set to trigger on the main flame peak. Setting the trigger level correctly prevents the hydrocarbon and sulfur responses from overlapping as the flame propagation slows in hydrocarbon-rich flames. The Sulfur response is proportional to the square of the Sulfur concentration in the flame whereas the Carbon response is directly proportional to the Carbon concentration. Therefore, when the amount of sample that is injected is doubled, the Sulfur-Carbon selectivity doubles (all other parameters being equal).
Phosphorus-to-Sulfur selectivity	• Install wide bore quartz combustor. • Use hydrogen as carrier gas. • Use *slightly* hydrogen-rich flame. Increasing Air1 flow until phosphorus response decreases by a factor of 2 will improve P/S selectivity greatly. • Adjust split valve such that detector is just out of tick-tock. • Set detector temperature ~300 °C. • Inject the smallest amount of sample consistent with adequate phosphorus signal. Sulfur interference is reduced in direct proportion to the reduction in sample size. For example, a 5 fold reduction in sample size will result in a 25 fold reduction in sulfur interference response, but only a 5 fold reduction in phosphorus response, for a net enhancement in selectivity of a factor of 5. • Decrease Gate Delay and Gate Width as much as possible while maintaining an acceptable Carbon selectivity. • Because Phosphorus emits sooner than Sulfur, a shorter Gate Delay and Gate Width improves the Phosphorus-Sulfur selectivity.

To Maximize ...	*Consider ...*
	• Additional phosphorus/sulfur selectivity can be achieved using the PFPD Analytical Software (03-925943-92). This option allows you to collect the full emission profile for the chromatogram and then post-run subtract out the sulfur response mathematically. For additional details contact your Varian representative.
Analytical throughput (decrease analysis time)	• Install wide bore quartz combustor. • Use hydrogen as the carrier gas at flow rates up to 10 mL/min. • Bracket only the region of the chromatogram that the PFPD can detect and for which the detector conditions have been optimized. **NOTE: Because the van Deemter curve for hydrogen is flat at very high flow rates, there is little loss of efficiency as the flow rate is increased to shorten the analysis time. The wide bore quartz combustor permits the use of higher flow rates, but this may not be a good choice if maximum Sulfur response is also the objective of the application.**
Effect of PFPD temperature	An initial detector temperature of 200 °C is recommended when optimizing the PFPD, but your sample may require a higher detector temperature to adequately separate and detect higher boiling sample components. The figure below shows the effect of increasing the PFPD temperature by 100 °C. The sample eluted from the column at a column temperature of 260 °C. At a detector temperature of 200 °C, the peak is broad with a slight shoulder. When the detector temperature was increased to 300 °C, the peak sharpened and the shoulder disappeared. The higher temperature also helps to assure uniform response for a wide range of samples.

To Maximize ...	***Consider ...***
	 

Establishing Tick-Tock

Tick-tock occurs when the combustible mixture ignites in the igniter chamber but does not propagate into the combustor chamber because the combustor has not yet filled with a combustible gas mixture. Optimum performance is generally achieved when the detector is operating near, but not in, the tick-tock mode.

Establishing Tick-Tock and subsequently the proper setting of the Air-Hydrogen needle valve is accomplished using either the scope mode of the PFPD Analytical Software or the procedure outlined below. The PFPD Analytical Software is available for desktop (03-925943-92) or laptop PCs (03-925943-91).

Follow these steps to adjust the gas flow rates for optimum performance of the PFPD. This adjustment should be done with the column oven at the highest temperature which will be used in the analysis. This will assure that the detector will remain in the normal operating mode throughout the column temperature program.

1. To monitor the detector baseline, connect a strip chart recorder or PC to the detector analog output. Turn off the AutoZero function (in the Detector section of the GC method). Set the Gate Delay to 4 msec and the Gate Width to 10 msec. Leave the trigger level at the default value, 200 mV. (This makes Tick-Tock easier to observe. Remember to switch back to the desired gate delay and width settings after setting the needle valve.)

2. Adjust the gas flow rates so that the PFPD is pulsing at about 2-4 Hz (pulses/second).

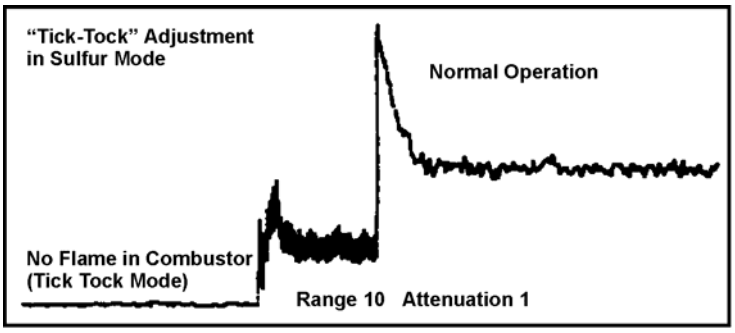
3. Adjust the Air-Hydrogen needle valve fully clockwise. This shuts off the flow of air-hydrogen to the combustor chamber while permitting the air-hydrogen mixture to pass around the outside of the combustor and enter the igniter chamber. The pulse rate should increase. As the flame stops propagating into the combustor, the detector baseline drops and becomes smoother (less noisy).

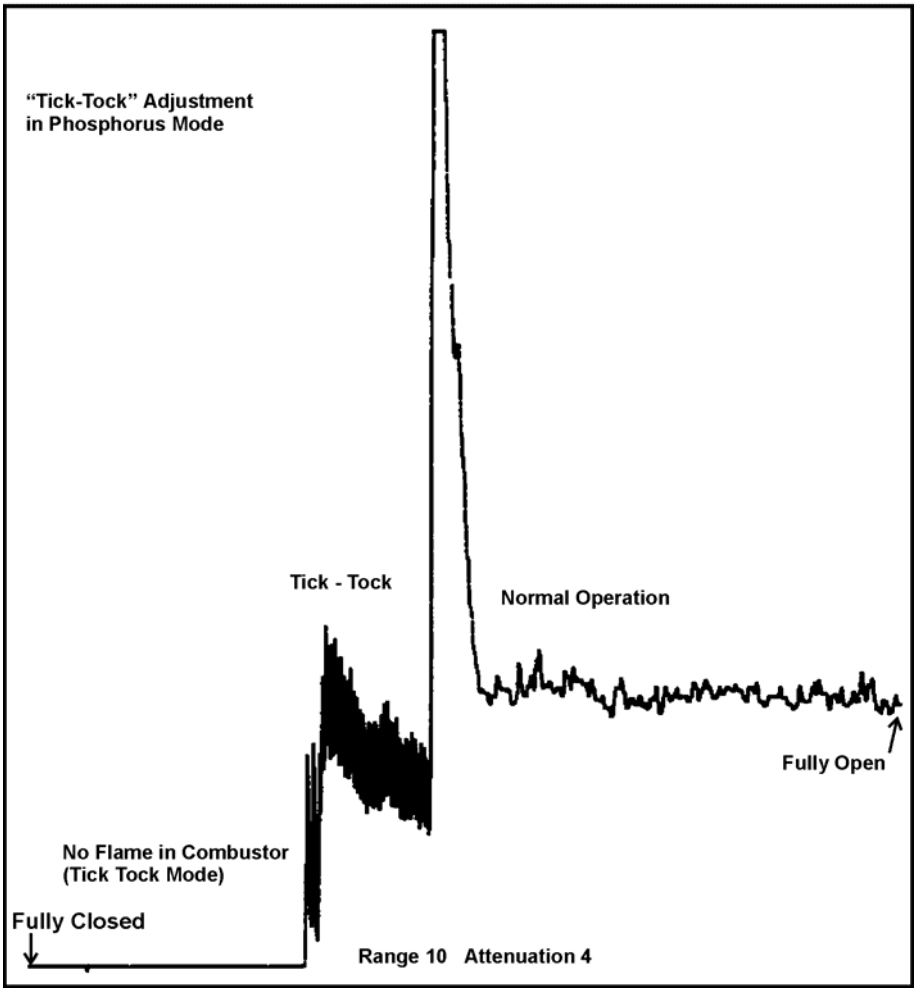
At low column flow rates (particularly with the 3 mm combustor), there may be enough diffusion of fuel mixture into the combustor chamber that the baseline is not completely smooth (noiseless).

4. Slowly turn the Air-Hydrogen needle valve counterclockwise. With each turn of the needle valve, more of the combustible mixture enters the combustor chamber. As the amount of combustible mixture entering the combustor chamber increases, the flame begins to propagate into the combustor chamber. First, the flame propagates into the combustor chamber on every third or fourth pulse and then, as the flow rate of the combustible mixture increases, on alternate pulses (tick-tock mode) and then finally on every

pulse. As you increase the flow rate of combustible mixture that enters the combustor, the baseline should rise sharply and become very noisy as the detector enters tick-tock. As the detector leaves the tick-tock mode, the baseline will rise and then fall and become less noisy. The pulse rate will also decrease as you pass through tick-tock, since more of the combustible mixture is burned on each pulse as you adjust the needle valve for normal operation.

5. Once the baseline reaches a steady value, you may adjust the needle valve an additional 1/2-turn counterclockwise to ensure stable operation of the PFPD. If you changed the gate delay and width, reset them to the proper values for your analysis.





Baseline noise changes as needle valve is opened.

Photomultiplier Tube (PMT) High Voltage

The PFPD detector is shipped with the electrometer and detector set up for the mode specified or the phosphorus mode, if not specified. The PMT (R647) high voltage is set at 510 volts typically for phosphorus. For the sulfur mode, the recommended voltage is somewhat higher, usually 550 volts. For the nitrogen mode, 580 - 620 volts is typical, since the R5070 PMT has a lower gain. This voltage range is also typical for S and P mode operation with the R5070 PMT.

Adjusting the Photomultiplier Tube (PMT) Voltage

The following procedure describes how to adjust the Multiplier Voltage when changing from one mode to the other, or to compensate for aging of the PMT over time.

1. If you have not already done so, adjust the detector gas flow rates for the mode of operation desired (Sulfur or Phosphorus). Use flow rate values that you determined previously or the recommended flow rates listed in the *Gas Flow Rates* table on Page 369.

2. When the detector is pulsing normally, press the *Detector,* select the PFPD tab,

3. If the Multiplier Voltage (PMT) voltage is:

   - Within 10-20 volts of the nominal value for the desired mode of operation, you may not need to make any further adjustment.

   - Not within this range, enter a new PMT voltage while observing the PFPD output on a strip chart recorder or on the Workstation System Control display.

4. If you want to optimize the detector sensitivity with respect to PMT voltage:

   - Set up the PFPD to operate in the desired mode. Set the detector to Range 10.

   - Slowly adjust the PMT setting while watching the baseline noise (displayed on a strip chart recorder or other output device).

- Starting at ~450 volts, increase the PMT voltage in increments of 20 to 50 volts while observing the peak to peak noise. At low PMT voltages, the baseline will rise with increasing voltage, but the peak to peak noise will remain unchanged. Continue increasing the PMT voltage until the peak to peak noise is approximately twice as great as it was at the lowest voltage. This PMT voltage should provide optimal detectivity and maximum dynamic range.

5. Note the PMT value displayed on the GC Detector Status display as above.

As the PMT ages, the voltage required to generate adequate sensitivity gradually increases. Replace the PMT when you can no longer achieve adequate sensitivity within the range of PMT available.

Optimizing the PFPD

The PFPD can be optimized in 2 ways:

- Repeated injections of standard.
- Continuous infusion of standard.

Tools and Equipment Needed

These tools are needed to complete the procedure for optimizing the PFPD using the method of repeated injections of standard. Refer to parts and supplies, on page 412.

- Flat tipped screwdriver.
- Printer/plotter or stripchart recorder.
- FPD test sample.
- 10 µL syringe.

Repeated Injections of Standard

Follow these steps to optimize the PFPD using the method of repeated injections of a standard.

1. Install the test column.

2. Set the Detector temperature to 200 °C for S mode, or 300 °C for P mode. Set Injector temperature to 200 °C for isothermal Injectors; 80 °C initial, 0 hold, program to 200 °C for 1079 PTV or 1177. Set column oven to 80 °C initial, hold 0.5 minutes, then program at 25 °C/minute to 200 °C and hold 2 minutes, or until the last peak of interest elutes.

3. Allow the chromatographic system to equilibrate for ~1 hour.

4. Turn the stripchart recorder or other display device ON.

5. Adjust the gas flow rates to those recommended for your carrier gas and flow rate in the Suggested Fuel Gas Flow Rates table on Page 369.

6. If necessary, adjust the *Air2* flow rate to give a flame pulse rate of ~2-4 pulses/second.

7. Inject 1.0 µL of the FPD Test Sample split ~20:1 if using a 1079 PTV or 1177 split injection. Inject 1 µL of a 20:1 dilution of the test sample in iso-octane if using a 1079 PTV or 1177 splitless or on-column injection or a 1041/1061 Injector.

8. Note the peak height of the sample peak eluting last, methyl parathion. When the GC is Ready again, increase the hydrogen flow by 20 digits on the counter, or 0.2-0.5 mL/min with Detector EFC inject the sample again and note whether the peak height increases. If so, continue to increase the hydrogen flow and reinject the sample until the peak height no longer increases.

   If the peak height does not increase by increasing the hydrogen flow, then decrease the flow by 20 digits on the counter, or 0.2-0.5 mL/min with Detector EFC and reinject the sample. Continue this procedure until the methylparathion peak is maximized.

Allow the system to equilibrate for ~2-3 minutes between flow rate adjustments so that the air and hydrogen filters are completely flushed with the new gas composition.

9. When the peak height is maximized, tune the fill time of the igniter volume to be just slightly greater than the fill time of the combustor volume. See Establishing Tick-Tock on Page 375.

Tools and Equipment Needed

These tools are needed to complete the procedure for optimizing the PFPD using the method of continuous infusion of standard:

- Printer/plotter or stripchart recorder.
- Sulfur diffusion source kit.

Continuous Infusion of Standard

Follow these steps to optimize the PFPD response using the method of continuous infusion of a standard.

1. Install the Sulfur diffusion source.

   Disconnect the column at the Injector end. Insert the sulfur permeation tube into the diffusion source holder, wrap the threads of the holder with Teflon thread sealant tape and attach the Injector adapter. Tighten the cap just enough to make a gas-tight seal.
   Insert an aluminum adapter seal in the end of the Injector adapter. Check that the crunch washer is properly seated at the bottom of the holder and not resting on the threads. Screw the Injector adapter-end of the diffusion source holder onto the Injector outlet fitting. Tighten the holder just enough to make a gas-tight seal.

If the permeation tube has not been used recently, set the Injector and column temperature to 50 °C and pass carrier gas over the permeation tube for ~1/2 hour. This decreases the surface concentration of the permanent.

2. Install the test column.

   If the test column is installed, shorten the length of column extending above the Injector nut to ~5 mm.
   Insert the column into the column-end of the diffusion source holder. Tighten the column nut as usual. The outlet of the holder uses the same fitting as the Injector outlet except that the holder requires an insertion depth of only
   1-2 cm.

3. Set the Injector and Column temperatures to 60 °C.

The concentration of standard that enters the detector is adjusted by increasing or decreasing the Injector and Column temperatures. However, 60 °C should be near optimum for the optimization procedure.

4. Adjust the column flow rate as required (~2 mL/min for the 320µ test column). Allow the chromatographic system and permeation tube and diffusion source holder to equilibrate for ~1 hour.

5. Adjust the gas flow rates to the recommended flow rates listed in the Gas Flow Rates table of Optimizing the PFPD on page 369. Set the detector range to 10. Set Autozero to OFF. Adjust the attenuation to keep the emission on scale.

6. If necessary, adjust the *Air1* and *Hydrogen* flow rates to give a flame pulse rate ~2-4 pulses/second. Alternately increase or decrease *Air1* and *Hydrogen* so that the flow rate remains approximately the same as the gas composition is changed. Allow the system to equilibrate for 2-3 minutes between flow rate adjustments so that the air and hydrogen filters are completely flushed with the new gas composition.

When adjusting the flow rate, turn the Air and Hydrogen flow controller dials in increments of 10 or 20, or make 0.2-0.5 mL/min adjustments using PFPD Detector EFC.

7. Increase the flow rate of *Hydrogen* by 10 dial units. Examine the emission on the stripchart recorder. Monitor the emission after each flow rate adjustment. If the sulfur emission increases, continue to increase the hydrogen flow rate until the response no longer increases. If the pulse rate increases beyond 4 Hz, reduce the *Air1* flow by 10 units and continue the procedure. Typically, noise is relatively independent of gas composition so that the relative gas composition can be adjusted with little or no effect on the noise.

Sulfur emission is maximized when the gas mixture is very rich in hydrogen.

8. Once the sulfur emission has been maximized, adjust the fill time of the igniter volume so that it is just slightly less than the fill time of the combustor volume. See Establishing Tick-Tock on Page 375.

9. Remove the column from the diffusion device holder, remove the diffusion device holder from the Injector outlet and reinstall the test column or the appropriate analytical column in the Injector.

Testing the PFPD Performance

The following procedure describes how to test the performance of the PFPD after it has been optimized for the desired detectivity and selectivity.

Tools and Equipment Needed

These tools are needed to complete the following procedure Refer to parts and supplies, on page 412.

- FPD test sample.
- 10 µL syringe.
- Test column, DB-1 320µ x 4M.

Follow these steps to test the chromatographic performance of the PFPD.

1. Configure the GC as described below:
   - Column: Test column (DB-1 320µ x 4M).
   - Injector: 1079 PTV or 1177.
   - Printer/plotter, stripchart recorder, or Workstation printout.
2. Select the mode (sulfur or phosphorus):
   - Turn the PFPD electrometer OFF.
   - Remove the GC Top-Covers.
   - Install the correct optical filter for the element of interest.
   - Set the Gate Delay and Gate Width for the element of interest.
   - Install a 2 mm quartz combustor and support for sulfur mode, or a 3 mm combustor and support for phosphorus.

   Replace the GC Top-Covers and turn the PFPD electrometer ON.
3. Set the GC method (or GC Workstation method) for the following bake-out conditions:
   - Oven temperature: 240 °C.
   - Detector temperature: 350 °C.
   - Injector temperature: 250 °C.

   Disconnect the test column, cap off the detector and operate the GC for at least 4 hours to bake out the column. Set the oven temperature to room temperature and wait until the oven and column are cool. Reconnect the test column to the detector.
4. Set the GC method (or GC Workstation method) for the following test conditions:
   - Injector: 200 °C isothermal or 80 °C initial, 0.00 hold, program to 200 °C for 1079 PTV modes.
   - Detector: Temperature 200 °C for S mode, 300 °C for P mode, Range 10, Autozero ON.
   - Column Oven: 80 °C initial, 0.50 min. hold, 25 °C/minute to 200 °C, hold 2.00 minutes, or until last peak of interest elutes.

   Set PMT voltage to 510V for phosphorus or 550V for sulfur. If you are using the R5070 (N mode) PMT, set voltage to 600V or use values obtained in on page 378.
5. Operate the GC for at least 1 hour to ensure that the PFPD and its electronics are thermally stable. Re-optimize the PFPD as recommended in Optimizing the PFPD on Page 368.

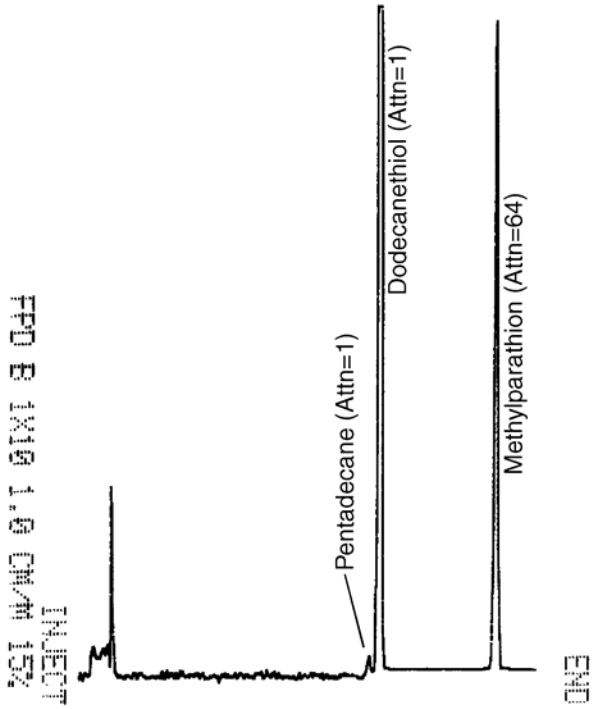
6. If you are using a split injection, inject 1 μL of FPD test mix with a split ratio of 20:1. If you are using a non-splitting Injector, dilute the FPD Test Sample 1:20 in iso-octane and inject 1.0 μL of the diluted FPD Test Sample.

7. The resulting chromatographic peaks should have similar retention times, heights and resolution to those shown below.

8. Are the peak retention times substantially different?
   - If Yes, adjust the column temperature and/or column flow rate and repeat steps 5-6.
   - If No, replace the test column with the column to be used in your particular application, adjust the GC conditions to appropriate values and proceed with the analysis.

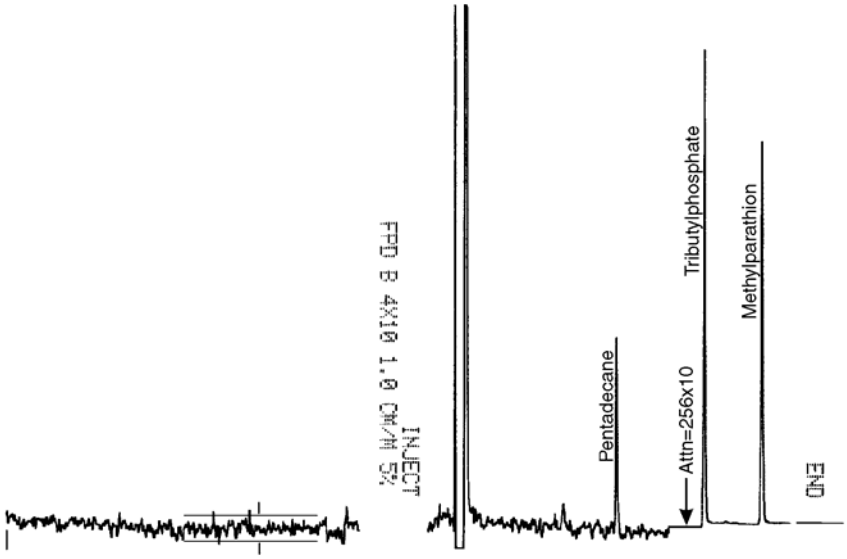
PFPD Test Chromatogram: Sulfur Performance

Refer to the figure below to check the chromatographic performance of the PFPD for sulfur. Compare the test chromatogram to the chromatogram shown below in terms of peak retention times, peak heights and peak resolution.



PFPD Test Chromatogram: Phosphorus Performance

Refer to the figure below to check the chromatographic performance of the PFPD for phosphorus. Compare the test chromatogram to the chromatogram shown below in terms of peak retention times, peak heights and peak resolution.



Detectivity

Refer to the instructions below to calculate the detectivity for sulfur and phosphorus with the PFPD:

- Determine the peak-to-peak noise (N_{p-p}) on the baseline. Collect at least 5 minutes of baseline noise at an attenuation which will allow easy measurement of the noise.
- Inject a known amount of standard (~100 x noise) and determine the peak height (H) for Sulfur or peak area (A) for Phosphorus. The instructions above will give a methyl parathion peak which contains 120 pg of both sulfur and phosphorus.
- Determine the width (W) of the peak at half-height ($W_{½}$) for phosphorus, or the width at 1/4 height for sulfur. Calculate detectivity (D) according to the formula below:
    - $D_{phosphorus} = [Wt \times (2 \times N_{p-p})]/[W_{½} \times H]$
    - $D_{sulfur} = [Wt/W_{¼}] \times SQRT\ [(2 \times N_{p-p})/H]$

Detectivity is expressed in units of weight/time. Wt is the weight of the specific element injected (e.g., weight of methyl parathion multiplied by 0.12 for either sulfur or phosphorus). $W_{¼} \cong 1.5 \times W_{½}$. The term SQRT means to take the square root of the product in brackets. If you are using peak areas or peak heights that have been calculated by a data system, use the counts-to-mm conversion factor from the data system.

PFPD Test Chromatogram: Sulfur Detectivity

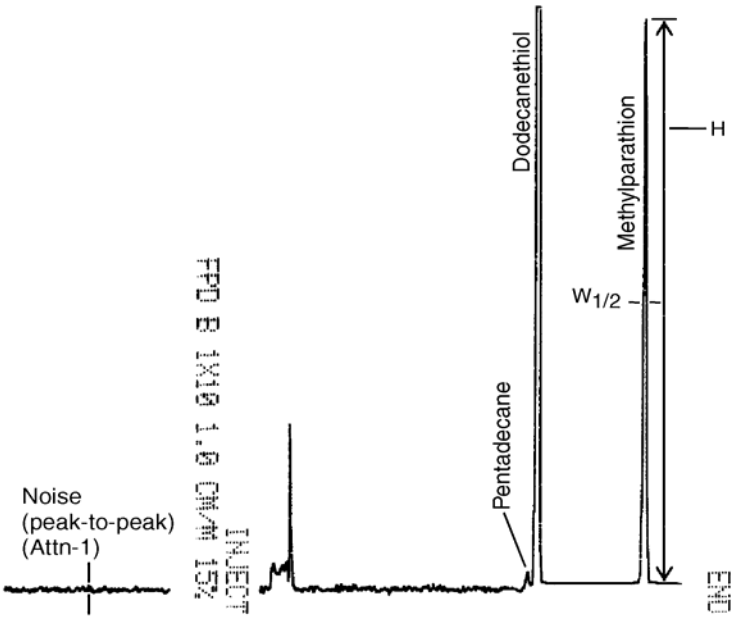
Refer to the figure below to check the detectivity of the PFPD for sulfur.

$$D_s = [W_s /(1.5 \times W_{1/2})] \times SQRT(2 \times N/H)$$

where D_s = Detectivity (pg/sec) for sulfur, W_s = Weight (pg) of sulfur, $W_{1/2}$ = width (sec) at half height of sulfur peak, N = peak-to-peak baseline noise (mm), H = height (mm) of sulfur peak, SQRT = square root

As an example, for the following test chromatogram: W_s = 120 pg, $W_{1/2}$ = 2.2 sec, N = 1 mm (1 mm x attenuation of 1), H = 4992 mm (78 mm x attenuation of 64) then

$$D_s = [120 \text{ pg} /1.5 \times 2.2 \text{ sec}] \times SQRT(2 \times 1 \text{ mm}/4992 \text{ mm}) = 0.73 \text{ pg/sec}$$



PFPD Test Chromatogram: Phosphorus Detectivity

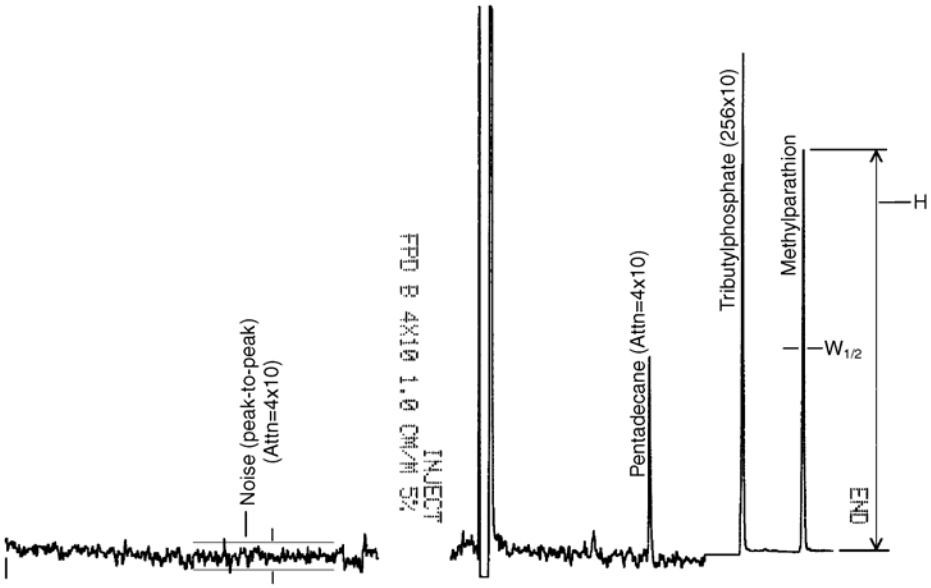
Refer to the following figure to check the detectivity of the PFPD for phosphorus.

$$D_p = [(W_p \times 2 \times N)/(H \times W_{1/2})]$$

where D_p = Detectivity (pg/sec) for phosphorus, W_s = Weight (pg) of phosphorus, $W_{1/2}$ = width (sec) at half height of phosphorus peak, N = peak-to-peak baseline noise (mm), H = height (mm) of phosphorus peak

As an example, for the following test chromatogram: W_p = 120 pg, $W_{1/2}$ = 2.5 sec, N = 16 mm (4 mm x attenuation of 4), H = 17152 mm (67 mm x attenuation of 256) then

$$D_p = [(120 \text{ pg} \times 2 \times 16 \text{ mm})/(17152 \text{ mm} \times 2.5 \text{ sec}) = 0.090 \text{ pg/sec}$$



Using the Gate Delay and Gate Width Parameters

Through the Gate Delay and Gate Width parameters you have access to the control functions of the gated integrator located on the PFPD electrometer board. These control functions enable you to select the time period (or region) of the emission spectrum that is to be integrated, thereby improving the sensitivity and selectivity of the PFPD.

What Can Be Controlled Through the Adjustments?

Three functions can be controlled through the Adjustments:

- Trigger Level: signal required to start (trigger) the delay and width timer.
- Gate Delay: time in milliseconds after triggering before integration of the detector signal begins.
- Gate Width: time in milliseconds after the Gate Delay during which the detector signal is integrated.

The appropriate values for the Gate Width and the Gate Delay are determined from the emission profile (time x emission intensity) of the element of interest. Appropriate values for a number of elements are listed in the following table.

Preset and Range of the Control Functions

Refer to the table below for a description of the preset values and ranges of the functions that can be adjusted.

Function	*Preset*	*Range*
Trigger Level	200 mV	10 - 2000 mV
Gate Delay	4 msec	0.5 - 20 msec
Gate Width	10 msec	0.5 - 20 msec

Table 58: Preset Values PFPD

For those users interested in gaining a more detailed understanding of the PFPD emission profile, the analog detector signal, the trigger state and the integrator gate state are available at test points on the edge of the electrometer board and on the pins of connector J4 on page 346.

When displayed on an oscilloscope, these signals allow detailed investigation of the emission time profiles of the flame background and of elements of interest, so that the detector parameters may be fine tuned for optimum selectivity and detectivity.

These signals may also be displayed and manipulated using the PFPD Analytical Software: 1) 450-GC and Desktop PC (03-925943-92) and 2) 450-GC and laptop PC (03-925943-91).

For more information on the use of these signals, please contact your Varian representative.

Setting the Trigger Level

Tools and Equipment Needed

These tools and equipment are needed to complete the following procedures.

Refer to parts and supplies, on page 412.

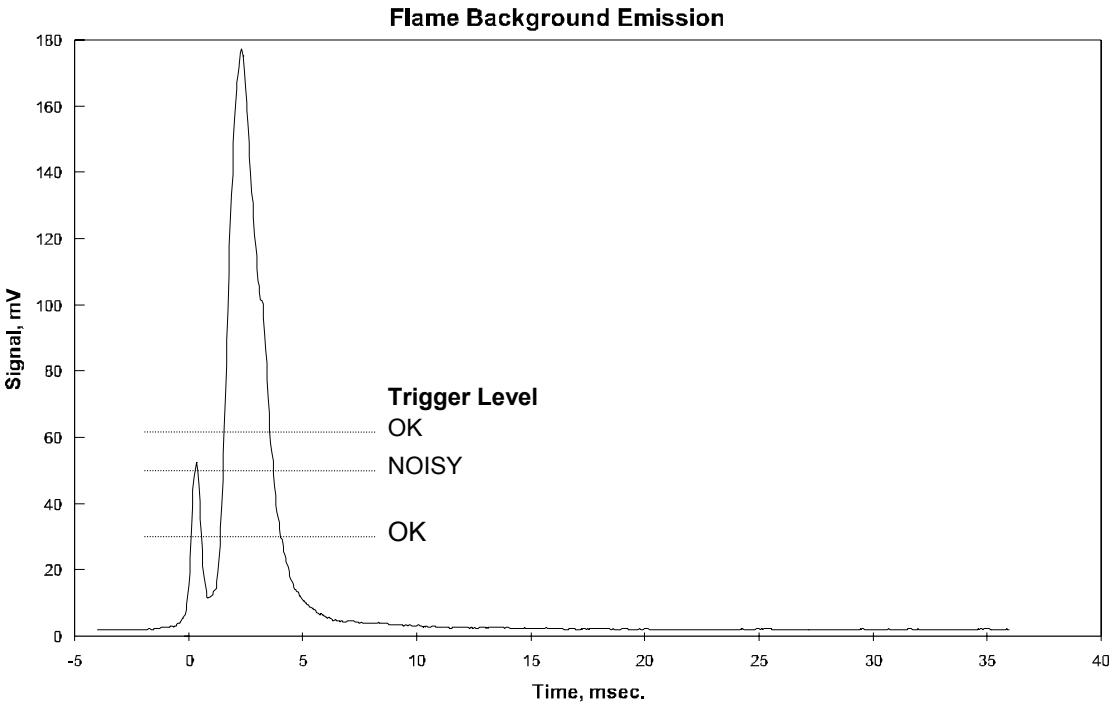
- Printer/plotter or stripchart recorder.
- FPD test sample.
- 10 µL syringe.

The trigger level is preset to 200 mV. You may need to set the trigger level lower if you are using a narrow band interference filter which does not transmit much of the flame background. You may need to set the trigger level higher than 200 mV for optimum operation with some elements which have no delayed emission, or very short emission delay times.

To set the trigger level, follow the steps below:

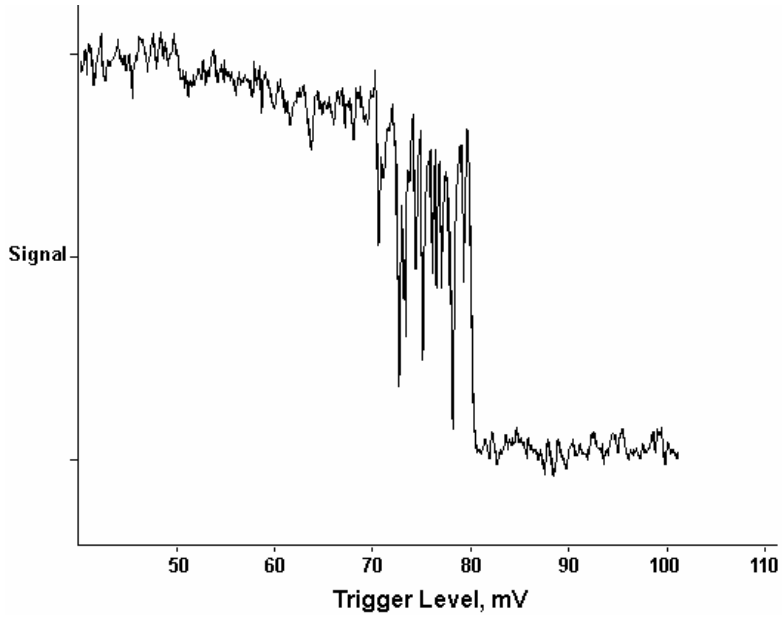
1. Press the Detector key and select the PFPD you wish to adjust. Turn OFF initial Autozero in the active method.

2. Press the Adjustments button to access the integrator parameters. Set Gate delay to 3.5 ms. This allows the tail of the prompt flame background emission to be integrated as a test signal. This figure shows a typical flame background emission profile.



3. Set the trigger level to the minimum and observe the noise on the detector output using a strip chart recorder or the Star Workstation status display. Gradually increase the trigger level. The signal level should decrease and the noise should also decrease. Select a trigger level which gives minimum noise. This will usually be triggering on the preliminary emission pulse.

4. If you will be detecting an element such as nitrogen, which emits very promptly after the main flame propagation, you may get better results by continuing to increase the trigger level until triggering is occurring on the main background emission pulse itself. As you increase the trigger level, the noise will increase substantially at one point. This is due to the trigger occurring sometimes on the apex of the preliminary emission pulse and sometimes on the main pulse. Increasing the trigger level 10 to 20% above this point should provide very stable triggering for detection of prompt emitting elements. This setting is also preferred when the effects of large amounts of hydrocarbons on emission timing must be minimized. The following figure illustrates the signal observed during such an adjustment.



Setting Gate Delay and Gate Width

The following table contains gate delay and gate width settings which are recommended for routine detection of common elements. Try these settings first when setting up your PFPD to detect these elements.

Element	*Gate Delay*	*Gate Width*
Sulfur	6	20
Phosphorus	4	10
Nitrogen	3.5	8

Table 59: Recommended Settings for common elements

Set the Gate Delay by Repeated Injections of Standard

The appropriate Gate Delay can be determined by injecting a standard and adjusting the Gate Delay until the required selectivity is achieved.

To set the Gate Delay by the method of repeated injections of standard, follow these steps:

1. Install the test column.
2. Set the Detector temperature to 200 °C for S mode, 300 °C for P mode. Set Injector temperature to 200 °C for isothermal Injectors; 80 °C initial, 0.00 hold, program to 200 °C for 1079 PTV mode. Set column oven to 80 °C initial, hold 0.50 minutes, then program at 25 °C/minute to 200 °C and hold 2.00 minutes, or until the last peak of interest elutes.
3. Allow the chromatographic system to equilibrate for ~1 hour.
4. Turn the stripchart recorder ON.
5. Adjust the gas flow rates to those recommended for your carrier gas and flow rate in the Suggested Fuel Gas Flow Rates table in Optimizing the PFPD (Page 369). The Air2 flow rate has little effect on the detectivity of the PFPD and can be set to 10 mL/min.
6. If necessary, adjust the Air1 and Hydrogen flow rates to give a flame pulse rate ~2-4 pulses/second.
7. Inject 1.0 μL of the FPD Test Sample split ~20:1 if using a split Injector. Inject 1.0 μL of a 20:1 dilution of the test sample in iso-octane if using a non-splitting Injector. Or, inject another sample of interest.

8. If the desired selectivity has not been achieved, increase the Gate Delay by 0.5 msec and inject the standard to check the effect of this change. Continue to increase the Gate Delay until the desired selectivity is achieved.

In the case of elements which emit very near the flame background peak, selectivity may be improved by opening the combustor flow needle valve somewhat beyond the position determined in the procedure Establishing Tick Tock. An increase of 1/4 to 1/2 turn counterclockwise is usually sufficient.

Set Gate Width

Tools and Equipment Needed

These tools and equipment are needed to complete the following procedures. Refer to parts and supplies, on page 412.

- Printer/plotter or stripchart recorder.
- FPD test sample or other standard.
- 10 µL syringe.

1. Set Gate Width to minimum
2. Inject an appropriate standard, calculate the detectivity for this Gate Width.
3. See: Testing the Performance of the PFPD on page 382 for instructions on calculating the detectivity of sulfur or phosphorus with the PFPD.
4. Increase Gate Width by 1 msec, inject standard, calculate detectivity.
5. Continue to increase Gate Width as long as detectivity continues to improve.

Building a GC Method

The procedure for building a GC method involves 4 tasks:

- Turn the PFPD electrometer on.
- Set the detector temperature.
- Set the range of the PFPD electrometer.
- Set the gated integrator parameters.

1. Press the Detector key and select the PFPD tab.
2. Turn the detector oven ON and set the desired detector temperature.
3. Turn the detector electronics ON.
4. Select the Range you wish to use and turn on initial Autozero if desired. You may also time program Range and Autozero functions if your analysis requires it.
5. Enter the Gate Delay, Gate Width and Trigger Level parameters as determined in the preceding procedures, or from the table provided.
6. If you wish to use the analog output from your detector, select one of the Analog Output ports, enter the position of the PFPD detector you are using (Front, Middle, or Rear) and enter an attenuation value. If the analog output is going to a data system or integrator, an attenuation of 1 is strongly recommended. If the output is going to a strip chart recorder, note that you may time program the attenuation of the analog output port.

Building a GC Workstation Method

The procedure for building a PFPD Detector section as part of a GC method involves 4 tasks:

- Turn on the PFPD electrometer.
- Set the detector temperature.
- Set the range of the PFPD electrometer.
- Set the Gated Integrator parameters.

1. Open the method to which you wish to add the PFPD section. Select the 450-GC section and select the Detector page. Select the PFPD tab.
2. Set the Electronics and Detector Oven to ON.
3. Set the Detector Temperature to an appropriate value for your analysis.
4. Set the Range and Autozero as required. Note that these parameters are time programmable.
5. Set the Trigger level, Gate Delay and Gate Width as required.
6. If you wish to use the Analog Output in addition to the digital signal used by the Star Workstation, select the Analog Output page, select the output channel you wish to use and select the PFPD position and attenuation.

Quantitation

Quantitation with the PFPD

When using the PFPD, it is important to understand how its pulsing flame can affect the accuracy and precision of a quantitative analysis.

As described previously, the combustor and igniter chambers of the PFPD fill with a combustible mixture of air and hydrogen. The combustor chamber fills with the combustible mixture at about the same time as the igniter chamber. When the gas mixture enters the igniter, the mixture ignites and the flame propagates back through the combustor and then extinguishes. The combustor then begins to fill with the gas mixture and the process repeats. The rate of this repetitive process can vary, but for optimum performance of the PFPD, you must adjust the gas flow rates in order to strike a balance between the fill time of the combustor chamber and the fill time of the igniter chamber. It is important that the combustor chamber fill slightly before the igniter chamber in order to ensure that the flame propagates completely back to the combustor chamber. However, the combustor chamber should not fill too fast (relative to the igniter chamber) or some of the sample exits the detector before it is combusted resulting in poorer detectivity.

Because the PFPD pulses, the emission derived from the PFPD appears as discrete signals separated in time. This requires electronics that are quite different from the conventional non-pulsed FPD in which the emission signal increases then decreases without interruption. That is, the PFPD electronics are not sampling discrete points on the eluting peak, as in the case of a conventional FPD. Rather, the PFPD stores the emission output derived during each pulse and generates a peak (or waveform) that may or may not be identical to the actual eluting peak shape, but which contains all the signal information from the chromatographic peak.

Given these unique properties of the PFPD, there are differences between quantitation based on the height or area of the chromatographic peaks.

Peak Area Quantitation

In general, quantitation based on peak areas is preferred because it is least affected by the pulsing nature of the PFPD and the way the PFPD electronics handles the resulting emission signal. Peak areas are best because once the fill rates of the PFPD are appropriately adjusted, any sample that enters the detector contributes to the resulting signal (i.e. all the sample that enters the detector between pulses is combusted), no matter how narrow the chromatographic peak.

Peak Height Quantitation

Quantitation based on peak height, on the other hand, is less precise and depends, somewhat, on the widths and resolution of the eluting peaks. Peak height is determined by the pulse which contains the maximum concentration of the eluting peak and to some extent is dependent on how well centered the concentration maximum of the eluting peak is relative to a pulse. The quantitative precision of the PFPD is not affected much by very narrow or very wide peaks. For very narrow peaks, almost the entire peak is combusted in one pulse and the resulting peak height is thereby equivalent to the total emission. For very broad peaks, the pulsed output follows the chromatographic peak shape. There may be some intermediate width peaks for which peak height precision is worse, but these cases can be addressed by changing the pulse repetition rate appropriately. For these intermediate width peaks, the peak is eluting during 2 or 3 pulses and the position of these pulses relative to the peak position can cause peak height to vary. Peak area is not greatly affected.

Peak Resolution with the PFPD

Higher repetition rates (up to 10 Hz) are necessary when you must resolve 2 closely spaced peaks. Higher repetition rates give the best possible peak shape definition (at a small cost in detectivity). However, peak resolution is not usually a problem at lower repetition rates since the number of possible sulfur or phosphorus containing peaks is fairly small at the beginning of a chromatogram where peak resolution is likely to be a problem.

Quantitation in the Sulfur Mode

Since the electronically excited S2 molecule is the emitting species in the sulfur mode, the signal generated by the PFPD is proportional to the square of the sulfur concentration in the combustor. Because it takes 2 sulfur atoms to form each excited S2 molecule, detectivity is improved by increasing the concentration of sulfur in the combustor by confining the emission to a smaller volume (i.e. 2 mm combustor).

Square Root Function

The PFPD electrometer (through a square root function) converts the quadratic response (sulfur emission varies as the square of sulfur concentration) to a linear response (sulfur signal varies as a direct function of the sulfur concentration) by taking the square root of the response. The square root function can be used for the determination of total sulfur in a hydrocarbon matrix, for example, where individual resolved peaks may not be present and a total area of sulfur response is needed.

Normal/Square Root setting

The *square root* mode is selected by the NORMAL/Square Root field. When Square Root is selected, the output from the PFPD is the square root of the sulfur emission. When NORMAL is selected, the output of the PFPD is not converted or adjusted. Use this NORMAL setting when operating the PFPD in the phosphorus mode and when the sulfur signal is to be processed by a data system using the square root of height.

Detectivity of the NORMAL Versus Square Root Modes

In the Square Root mode, the amplifier signal is applied to the input of a square root circuit which produces an output equal to 10 times the square root of $V_{in}/10$. This output is linear with sulfur concentration but by the nature of the square root function is noisy. Detectivity in the hardware Square Root mode is often 2 to 4 times worse than in the Normal mode under the same detector conditions.

How to Minimize Quenching of Sulfur Response

When a large amount of hydrocarbon coelutes with a sulfur compound of interest, the sulfur response may be quenched. This quenching is the result of a reaction of CO, formed by combustion of the hydrocarbon, with S atoms. In some cases this quenching can be avoided by use of a more selective column to eliminate the coelution, e.g., COS/propylene, benzene/thiophene, toluene/methylthiophene. Use of a 3 mm ID combustor with somewhat increased Air1 flow may also help to reduce quenching. Finally, injection of the smallest amount of sample which gives an adequate signal to noise ratio will minimize quenching.

Advanced Operation

This section describes the operation of the PFPD in the Nitrogen mode, including:

- Theory of operation, describing the basis for nitrogen detection and including suggested gas flow rates.
- Installation of the nitrogen mode parts - for users who are adding the nitrogen mode capability to an existing PFPD. Includes an exploded view of the parts.
- Nitrogen mode operation - provides some suggestions for enhancing or modifying nitrogen mode performance.
- Phosphorus mode/Sulfur mode operation - describes some changes which may be required to detect phosphorus or sulfur using the nitrogen mode PMT.
- Spare Parts and Supplies.
- Additional Elements - describes preliminary operating conditions for the detection of the following elements: Arsenic, Selenium, Tin and Manganese.

Theory of Operation

Nitrogen mode is based upon combustion of nitrogen containing compounds to form NO. NO then reacts with hydrogen atoms remaining in the combustor after the flame is extinguished to form HNO*, an electronically excited molecule which emits light in the 700 - 800 nm range of the near infrared. Because these emission wavelengths are beyond the range of the photomultiplier tube used in the standard PFPD, the Nitrogen mode requires a new red-sensitive photomultiplier and PMT holder hardware, along with an appropriate dark red colored glass filter.

Because N mode is based on complete combustion of the sample molecule to form HNO*, the PFPD response is much less dependent on the chemical nature of the sample than is the TSD response, which depends on the presence of a C-N bond in the molecule. Thus even inorganic nitrogen compounds such as ammonia, hydrazine and nitrogen oxides are detectable by the N mode PFPD, whereas these compounds give no response on the TSD. The only nitrogen compound known to give no useful response on the PFPD is molecular nitrogen, N_2. Compounds such as nitroglycerin and other nitrate esters, nitroaromatics and nitroso compounds give a somewhat enhanced response when compared to more reduced nitrogen compounds, but the enhancement amounts to only ~20%, so for most purposes the N mode PFPD can be considered to give a uniform response to all nitrogen compounds, as it

does for sulfur and phosphorus compounds. Uniformity of response may be enhanced by increasing the oxygen flow into the combustor, at the cost of some reduction in sensitivity.

Oxidation of nitrogen requires a hotter flame than either phosphorus or sulfur. This is reflected in the following table of recommended flame gas flow rates. In general, the nitrogen mode requires a more air rich flame and suffers a larger reduction in sensitivity as carrier flow rate increases. Use of the 3 mm combustor provides best detectivity.

Table of Optimum Flow Rates

		Optimum Flow Rate, mL/minute		
Carrier Gas	***Flow Rate, mL/minute***	***Air1***	***Hydrogen***	***Air2***
Hydrogen	2	12.5	7	15
	5	12.5	8	15
	10	28	3	15
Helium	2	14	14	15
	5	17.5	20	15
	10	17	17	15
Nitrogen	2	15.5	18	15
	5	18	19	15

Table 60: Optimum Flow rates PFPD

Installation of the Nitrogen Mode Parts

If you are upgrading a Phosphorus/Sulfur PFPD to add the Nitrogen mode, you will need to replace the photomultiplier tube housing and filter currently installed on your PFPD with the PMT assembly and filter supplied with your upgrade kit. Refer to the figure below for identification of the components of the N mode assembly. The steps involved are:

1. Turn off the PFPD. Alternatively, turn off GC power.

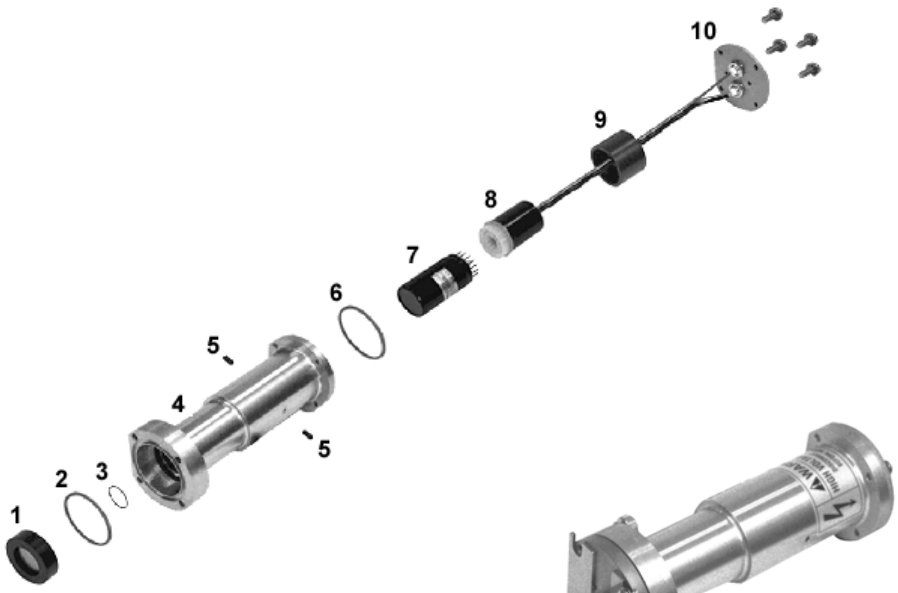
2. Disconnect the high voltage and signal cables from the base of the P/S PMT housing.

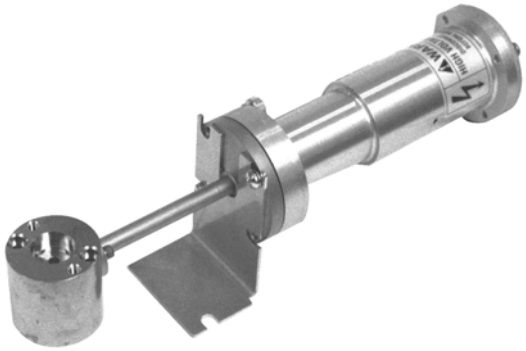
3. Remove the PMT housing by removing the 2 thumb screws securing the housing to the light pipe flange. Store the P/S PMT housing so as to protect it from mechanical damage and bright light.

4. Insert the Nitrogen mode filter in the recess in front of the PMT in the N mode housing. Be sure the silicone O-ring is in its place in the groove on the face of the housing flange. Using the 2 screws supplied with the upgrade kit, attach the N mode PMT housing to the light pipe flange. Be sure that the O-ring makes a light-tight seal.

5. Reconnect the signal and high voltage cables to the connectors on the base of the N mode PMT housing. Restore power to the PFPD.

6. Press the Detector key, select the PFPD and press the Adjustments soft key. Set a Gate Delay of 3.5 msec, a Gate width of 8 msec and a PMT Voltage of 600 volts.





Item	Description
1	Filter Mount
2	O-ring
3	O-ring, PMT Mounting
4	PMT Housing
5	Set Screws
6	O-ring
7	PMT
8	Socket
9	Retainer
10	Connectors with Mounting Plate

Nitrogen Mode Operation

Operation in the nitrogen mode is very similar to the sulfur and phosphorus mode operation described in detail elsewhere in the manual. The following differences should be noted:

1. The nitrogen mode uses a Hammamatsu R5070 red sensitive photomultiplier tube. This PMT may be operated at a somewhat higher voltage than the standard phosphorus/sulfur photomultiplier. A typical voltage of 600 volts has been used for the nitrogen mode.

2. Nitrogen emission duration is similar to but somewhat shorter than phosphorus emission, occurring over a period of up to ten milliseconds. A good starting point for Nitrogen mode tuning would be a gate delay of 3.5 milliseconds and a gate width of 8 milliseconds.

3. The Nitrogen mode requires somewhat finer control of the needle valve, since the best sensitivity is achieved just after the tick tock mode of operation is corrected. The needle valve should be opened only enough to bring the detector out of tick tock and reduce the background noise to a usable level. Then evaluate the signal to noise ratio of a nitrogen standard while adjusting the Gate Delay until the optimum signal/noise is achieved.

4. The optimized flame gas flow rates in the table above give the best overall detectivity for all nitrogen compounds, with somewhat enhanced response from oxidized nitrogen species. If you require more uniform response regardless of nitrogen compound type, you may increase the Air1 flow by 10 to 20%. This will enhance the response of reduced nitrogen relative to oxidized nitrogen, at the expense of some loss in overall detectivity. We have found a mixture of 2-nitrotoluene, 2,6-di-t-butylpyridine and N,N-diethylaniline useful as a probe of response uniformity.

5. When properly tuned, the Nitrogen mode PFPD shows no response to simple alkanes at fluxes up to about 1 microgram/second hydrocarbon. Above this flux the response increases until flame propagation is completely inhibited at about 30 micrograms/second. Selectivity against hydrocarbon interference can be increased by opening the needle valve 1/2 to 1 turn beyond the optimum for detectivity.

6. The near-IR sensitive PMT may respond to emissions from the detector itself at very high detector temperatures. If you need to operate at temperatures above 320 °C, you may need to increase the trigger level to be sure that the trigger point is well above the near-IR background. You may also find that addition of a short pass filter which cuts off wavelengths longer than those of your element of interest will improve detectivity and dynamic range. Optimum detector temperature for most nitrogen compounds is about 250 °C.

Parts and Supplies (Nitrogen Mode)

In general, the parts and supplies needed to operate the N mode PFPD are the same as those used with the P/S mode PFPD. The following are additional parts which may be required.

Part Number	*Description*
03-925119-01	Filter Mount with Nitrogen Mode (RG9) filter.
03-925119-02	Filter Mount with Phosphorus mode (GG495) and Red Absorbing (BG39) filters.
03-925128-00	Photomultiplier tube, Nitrogen mode.

Phosphorus Mode Operation

Operation in the phosphorus mode is the same with the Nitrogen mode PMT as it is with the standard PMT. To eliminate interference of nitrogen in the detection of phosphorus, a Schott BG39 red blocking filter may be used in conjunction with the Schott GG495 phosphorus filter. The PMT voltage may be run slightly (~20-50 volts) higher than the standard PMT.

Sulfur Mode Operation

No changes in operation are required to use the Nitrogen mode PMT in the Sulfur mode. The PMT voltage may be run slightly (~20 - 50 volts) higher than the standard PMT.

Options to Aid Optimization with Other Elements

Two options which may prove very helpful in developing optimum conditions for the detection of additional elements are the ChromatoProbe Kit (03-925677-91) and the PFPD Analytical Software (03-925943-92 for desktop PCs or -91 for laptop PCs). The ChromatoProbe, used in conjunction with a 1079 PTV Injector, allows the introduction of a controlled, continuous flux of the element of interest into the PFPD by vaporization of a volatile compound of the element from a temperature-controlled cup held in the 1079 PTV Injector. The PFPD Analytical Software displays the time profile of emissions on a pulse by pulse basis on the PC screen and allows post-run optimization of gate delay and gate width for maximum signal to noise ratio for any desired element. Please consult your Varian representative for more information on these options.

Multi-element Detection

In some applications, it may be desirable to detect more than one heteroatom simultaneously. Several combinations which may be of interest are: Sulfur plus phosphorus mode – use a WG345 filter with the standard PMT or WG345 + RG39 filters with the R5070 PMT if nitrogen interference is undesirable. Use a gate delay of 4 msec and adjust gate width to balance sulfur and phosphorus response.

Sulfur + phosphorus + nitrogen mode – WG345 filter with R5070 PMT. This combination will also detect Sn, Se and As.

Sulfur + Nitrogen mode – R5070 PMT with BG3 filter for suppression of phosphorus response.

Arsenic Selective Detection

The following recommendations are based upon work reported by Prof. Aviv Amirav and Hongwu Jing and are accurate to the best of our knowledge. This is an area of ongoing research, so you may find that your application requires modification of some parameters.

Emission Wavelengths

Arsenic exhibits a very broad emission spectrum, extending from 360 to 800 nm. The choice of the wavelength at which to observe As emission is determined largely by the emission characteristics of interfering elements.

Emission Timing

Arsenic emission is longer delayed than sulfur, with a correspondingly longer emission duration. Use the Gate Delay and Gate Width recommended for sulfur, or increase delay even further for additional selectivity against nitrogen and phosphorus. A 3 mm combustor provides best sensitivity.

Flame Composition

Delayed arsenic emission is optimized in hydrogen rich conditions similar to those used for sulfur. Arsenic may also be detected in an air-rich flame, as described below.

Filters

Arsenic may be detected at a variety of wavelengths, depending upon the requirements of the analysis. Good detectivity (~6 pg/second As) and selectivity are achieved using an RG9 (nitrogen) or RG695 filter with the Nitrogen mode R5070 photomultilplier tube. Interference by P and N compounds can be reduced by using a long gate delay time. Tailing of the delayed emission can be reduced by operating the detector at the highest practical temperature, or by operating below 200 °C.

An alternative approach to arsenic detection uses an air-rich flame, a GG495 (phosphorus) filter and a very short gate delay. This air-rich mode gives very good detectivity, ~3 pg/second As. and very good selectivity against sulfur interference, but at the expense of reduced selectivity against hydrocarbon interference. This air-rich mode also eliminates the tendency to peak tailing observed with the hydrogen-rich flame emission.

Linearity and Dynamic Range

Arsenic response is linear. No information is available on linear dynamic range, although it is expected to exceed 10^3.

Other Considerations

Arsenic detection provides the most opportunities for optimization of any of the common PFPD-detectable elements. Selectivity can be optimized by the choice of air-rich or hydrogen-rich flame conditions, by the choice of gate delay and by the choice of optical filter. In some applications, it may even be desirable to reduce the selectivity among heteroatoms and instead look to selectivity only against hydrocarbons. For example, the ability to detect simultaneously arsenic, phosphorus, sulfur and nitrogen, while totally rejecting hydrocarbon response, is extremely important in the detection of chemical warfare agents for treaty verification purposes.

Selenium Selective Detection

The following recommendations are based upon work reported by Prof. Aviv Amirav and Hongwu Jing and are accurate to the best of our knowledge. This is an area of ongoing research, so you may find that your application requires modification of some parameters.

Emission Wavelengths

Selenium exhibits a fairly broad emission spectrum in the PFPD, extending from ~360 to ~550 nm, attributed to emission of the Se_2 molecule.

Emission Timing

Selenium emission is somewhat longer delayed than sulfur, with a correspondingly longer emission duration. Use the Gate Delay and Gate Width recommended for sulfur.

Flame Composition

Selenium emission is optimized in cool, hydrogen rich conditions similar to those used for sulfur. Since emission is due to Se_2, use the 2 mm combustor and keep all gas flows as low as practical to minimize dilution of the selenium concentration in the flame.

Filters

Selenium may be detected using either the BG12 (sulfur) or the GG495 (phosphorus) filter. Best detectivity, ~10 pg/second Se, is achieved using the BG12 filter, but this also gives the most interference from sulfur compounds. Detectivity with the phosphorus filter is ~20 pg/second Se, but sulfur interference is considerably reduced. Either filter gives essentially complete specificity against hydrocarbon interference.

Linearity and Dynamic Range

Selenium response is quadratic, like the sulfur response and can be treated by the same approaches used for sulfur signal processing. Dynamic range is approximately 10^3 for a given electrometer range.

Other Considerations

Since selenium emission depends upon recombination of Se to form electronically excited Se_2 molecules, use the lowest detector temperature which gives satisfactory chromatographic peak shapes. Selenium also exhibits a prompt emission which may be used to distinguish it from sulfur.

Tin Selective Detection

The following recommendations are based upon work reported by Prof. Aviv Amirav and Hongwu Jing and are accurate to the best of our knowledge. This is an area of ongoing research, so you may find that your application requires modification of some parameters.

Emission Wavelengths

Tin exhibits a fairly broad emission spectrum in the PFPD. A broad band between ~380 and 540 nm is attributed in the literature to interaction between a tin species and the quartz surface. The sharper bands at 610 and 695 nm have been attributed to SnH emission. The relative and absolute amplitudes of these bands may be a function of the PFPD flame composition.

Emission Timing

Tin emission is somewhat longer delayed than phosphorus and emission duration is intermediate between phosphorus and sulfur. Use the phosphorus gate delay and width for best sensitivity; use the sulfur gate delay and width for best selectivity against carbon interference.

Flame Composition

Tin emission is optimized in hydrogen rich conditions similar to those used for phosphorus or sulfur. Increasing Air1, Air2 and hydrogen by 50 - 100% each will help to reduce peak tailing. Either the 2 or 3 mm combustor may be used, with a slight preference for the 3 mm.

Filters

Tin may be detected using either the BG12 (sulfur) or the OG590 filter. Best detectivity, ~100 fg/second Sn, is achieved using the BG12 filter, but this also gives the most interference from high levels of sulfur compounds. Detectivity with the OG590 filter is ~1 pg/second Sn, but sulfur response is considerably reduced. Response with the OG590 filter is best with the red sensitive R5070 photomultiplier tube. A 610 nm interference filter has also been found useful in conjunction with the R5070 PMT.

Linearity and Dynamic Range

Preliminary results indicate that the tin response is linear and extends over more than three orders of magnitude.

Other Considerations

Tin compounds exhibit tailing in the PFPD, probably due to limited volatility of the tin combustion products. This tailing can be reduced by using higher gas flow rates and by raising the detector temperature to 350 °C to help volatilize the tin compounds. You may also find it helpful to insulate the detector tower using the PFPD tower cover, or by wrapping the tower with fiberglass cloth, being careful not to cover the outlet.

An alternative approach to tin tailing is to reduce the detector temperature as much as possible without impairing the chromatography. This reduces the “tail” amplitude, but spreads it out in time. Several minutes may be required for the detector to clean itself of tin deposits. Refer also to: J. A. Jacobsen, F. Stuer-Lauridsen and G. Pritzl, Organotin Speciation in Environmental Samples by Capillary Gas Chromatography and Pulsed Flame Photometric Detection (PFPD), Appl. Organometallic. Chem. 11, 737-741 (1997).

Manganese Selective Detection

The following recommendations are based upon work reported by Prof. Aviv Amirav and Hongwu Jing and are accurate to the best of our knowledge. This is an area of ongoing research, so you may find that your application requires modification of some parameters.

Emission Wavelengths

Manganese exhibits a strong atomic emission line at 403 nm, along with other weaker lines and bands in the 380 - 600 nm range. The 403 nm line is the only emission of analytical interest with the PFPD.

Emission Timing

Manganese shows no delayed emission, as expected for atomic emission. Use the minimum delay time and a gate width of 2 to 4 msec, depending on your flame propagation rate.

Flame Composition

Manganese emission is favored by a hot flame, which is achieved by a stoichiometric or slightly air rich air1/hydrogen mixture and minimum dilution of the flame by carrier gas flow. The manganese emission is not very sensitive to the exact gas composition, so you may vary the composition somewhat to enhance selectivity against other elements. Emission is enhanced by the use of the 3 mm ID combustor.

Filters

Because the manganese emission is not separated in time from the flame background emission, a narrow band interference filter is required to provide adequate selectivity against hydrocarbons and other interfering elements. A 404.7 nm interference filter with 10 nm bandwidth was used in Prof. Amirav's work. This filter is commonly available as part of a set of filters for the isolation of mercury emission lines and may be obtained from many sources of interference filters. Using this filter, detectivity of ~100 fg Mn/second and a selectivity against hydrocarbons of ~104 have been observed by Prof. Amirav.

Linearity and Dynamic Range

Manganese response is linear. No information is available on linear dynamic range, although it is expected to exceed 103.

Other Considerations

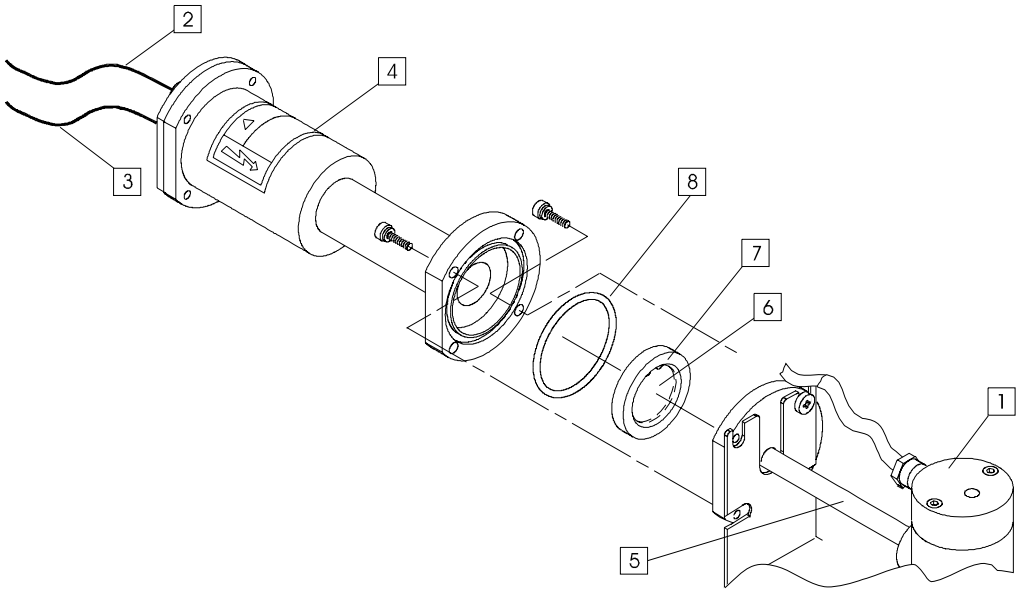
Manganese containing peaks show some tailing due to incomplete clearance of the manganese from the combustor. This tailing is minimized using near-stoichiometric flame conditions. It may be further reduced by using a detector temperature below 225 °C, but this may result in long term buildup of manganese deposits in the combustor, although this was not observed by Prof. Amirav.

Maintaining the PFPD

Replacing the Optical Filter

The choice of optical filter depends on the detection mode that is to be used.

- Sulfur mode: Use the BG-12 deep-violet glass filter.
- Phosphorus mode: Use the GG-495 yellow glass filter.
- Sulfur + Phosphorus mode: Use the optional WG-345 glass filter.



Item	*Description*
1	Ignitor cap
2	Photomultiplier tube (PMT) high voltage cable
3	Photomultiplier tube (PMT) signal cable
4	Photomultiplier tube (PMT) housing
5	Light pipe assembly
6	Glass filter
7	Filter holder
8	O-ring

Tools and Equipment Needed

These tools are needed to complete the following procedure:

- Phillips-head screwdriver.
- Glass filter in filter holder.
- Lab tissues.
- Capillary pick.

Disassemble the PFPD and Remove the Optical Filter

1. Turn OFF the PFPD electronics.
2. Remove the Detector cover from the top of the GC.
3. Remove the 2 thumbscrews holding the PMT housing (Item 4) to the light pipe assembly (Item 5). Carefully remove the PMT housing from the detector tower. The filter (Item 6) is loosely held in the filter holder (Item 7) that is recessed in the end of the PMT housing.
4. Invert the PMT housing over a clean lab tissue and allow the filter holder to drop out of the recess in the end of the PMT housing. If the filter holder should stick in the PMT housing, free it by using gentle pressure with a capillary pick or other clean utensil. Place this filter on a clean surface (e.g., lab tissue) or in its storage container.

Handle the filter holder by its outside edge. Do not touch the optical surface of the filter.

Replace the Optical Filter

1. If you are **cleaning the optical filter**, remove the C-clip holding the filter in the filter holder. Moisten a clean lab tissue with methanol or iso-propanol. Hold the filter by its outer edge and gently wipe both surfaces of the filter with the lab tissue. Wipe away the excess solvent with a clean dry lab tissue. Place the filter in the filter holder and reinstall the C-clip.
2. Hold the filter that is to be installed by its outside edge and slide it into the recess in the end of the PMT housing. Check that the filter is correctly seated so that its surface is flush with the rim of the PMT housing and the O-ring (Item 8) is properly seated in its groove.
3. Carefully join the PMT housing to the light pipe assembly and install and tighten the 2 thumbscrews.

Change the Detection Mode and Reassemble the PFPD

1. Go to the Adjustments page of the PFPD section.
2. Adjust the Gate Delay and Gate Width to correspond with the emission profile of the element(s) you wish to detect.
3. Turn on the PFPD electronics.
4. Go to ***Optimizing the PFPD*** on page 368 for instructions on optimizing the performance of the PFPD with the optical filter just installed.

Changing the Combustor Size

Although the PFPD can accommodate both narrow and wide bore quartz combustors, either quartz combustor will work adequately when operating in either the Sulfur or Phosphorus mode. If detectivity is not critical, you can switch between the 2 modes without changing the size of the quartz combustor. However, if you require the maximum detectivity for an analysis, the choice of the appropriate diameter (narrow bore or wide bore) of quartz combustor depends on the detection mode that is to be used.

- Sulfur mode: Use the narrow-bore (2 mm ID) quartz combustor for sulfur, selenium, or tellurium detection.
- Phosphorus mode: Use the wide-bore (3 mm ID) quartz combustor for all other elements.
- S + P mode: Use the narrow-bore (2 mm ID) quartz combustor to enhance S response. Use a 3 mm ID combustor to enhance P response or when quenching of S response is a problem.

For column flow rates over 5 mL/min, use the wide bore quartz combustor.

Proceed as on page 363.

Replacing a Dirty Combustor

The quartz combustor can become fouled after prolonged use, particularly with samples containing high levels of organometallics. However, the PFPD has a remarkable ability to clean itself of many deposits. Therefore, before replacing a combustor, try baking the detector at high temperature with normal pulse rate overnight or longer. If this does not restore expected noise and detectivity levels, proceed as described on page 363.

Ordering Parts and Supplies for the PFPD

To order parts or supplies for the PFPD, refer to the information below:

- In the USA and Canada, call 800-926-3000.
- In Europe, call your local Country office.
- Or, call your local Varian representative.

Description	***Part Number***
1/16" No-Hole Ferrule	SG0726841
FPD test sample	82-005048-03
Test Column, DB-1 320µ x 4M	03-912805-99
10 µL Syringe	47-000007-00
Permeation Tube, Sulfur	03-925195-00
Diffusion Source Holder	03-925196-00
Diffusion Source Kit, Sulfur	03-925157-00
PFPD Accessory Kit	03-925190-90
Spring	03-949786-00
Injector Adapter Cap	03-925200-00
450-GC PFPD High Voltage Cable	03-925490-01
Igniter Assembly with Cable	03-925461-01
Crunch Washers (Aluminum)	15-003347-00
Adapter Seal (Aluminum)	03-925132-00
O-ring, 1.487" ID x 0.103" Wall, Silicone	27-402826-00
Combustor, Quartz (2 mm ID, Sulfur)	03-925176-00
Combustor, Quartz (3 mm ID, Phosphorus)	03-925177-00
Combustor Support (2 mm ID, Sulfur)	03-925178-00
Combustor Support (3 mm ID, Phosphorus)	03-925179-00
Combustor Support seal (Aluminum)	03-925138-00
Combustor Support Wrench	03-925192-00
Allen Wrench, 9/64" (cleaned)	03-925194-04
Teflon Pipe Thread Tape	88-189610-00
Reducing Union (flow check)	16-000327-00
Teflon Combustor Extraction Tool (wide bore, 3 mm ID)	03-925180-00
Teflon Combustor Extraction Tool (narrow bore, 2 mm ID)	03-925181-00
Teflon Tweezers	29-898500-00
Tungsten Ceramic Scorer	01-900158-00

Description	***Part Number***
Varian Intelligent Digital Flowmeter	01-900115-00
Soap Flowmeter	03-949846-00
Magnifying Lens	00-997369-00
Filter Assembly, Sulfur	03-925151-01
Filter Assembly, Phosphorus	03-925151-02
Filter Assembly, Tin (Sn)	03-925151-03
Filter Assembly, Sulfur+Phosphorus	03-925151-04
Filter Assembly, Arsenic	03-925151-05
Filter, Sulfur + Phosphorus	03-925158-00
Screw, Detector Tower (8-32 x 1-1/2 SHCS)	03-925194-02
Screw, Igniter Adapter and Column Guide (8-32 x 3/4 SHCS)	03-925194-01

Troubleshooting

Overview

The following section describes how to troubleshoot and correct problems with the Pulsed Flame Photometric Detector (PFPD).

Using the Troubleshooting Guide

This troubleshooting guide outlines typical problems that you might encounter during the routine operation of the PFPD. To use the guide, first identify the symptom or problem (e.g., the PFPD flame does not ignite). Then, locate the problem in the following guide. Likely causes as well as instructions for correcting the problem are listed.

The troubleshooting guide also refers you to other sections of the Manual that may be helpful. Should you have questions when troubleshooting the PFPD, contact a Varian Customer Service Representative.

Flame Does Not Ignite

Possible Cause	*Solution*
Gas flow rates are too low or off.	Check that the carrier, air and hydrogen regulators read correct pressures. Check that air and hydrogen are flowing through the PFPD at the expected flow rates.
PFPD electrometer is OFF.	Turn the PFPD electrometer ON.
Detector tower too cold.	Be sure that detector oven power is On and allow enough time for the tower to approach the operating temperature. This is particularly important with very hydrogen rich conditions.
Igniter coil is broken.	Use a mirror to check that the igniter is glowing. If the coil is not glowing, the igniter coil may be broken. Disconnect the igniter cable from the electrometer board. Check for continuity. Replace the igniter coil and assembly if defective.
Detector is leaking gas from around washers and/or seals.	Check whether gas is leaking from around the aluminum crunch washers or the light pipe seal.
Air:hydrogen gas composition is incorrect.	If the igniter is glowing dimly, the gas composition may be too rich in hydrogen. To adjust the air:hydrogen composition, proceed as follows: • Note the current hydrogen dial setting, then turn the hydrogen flow rate OFF • Check that air flow rates are approximately: Air1 = 10-15 mL/min., Air2 = 15 mL/min. Reset the hydrogen flow to the correct setting.
Column is installed incorrectly.	Check whether the column is leaking or is improperly secured. Install the column correctly.

Possible Cause	*Solution*
Carrier gas flow rate is too high.	This is a particular problem when using helium as a carrier gas, as helium tends to cool the flame excessively, preventing the flame from propagating into the combustor. Decrease carrier gas flow rate to an appropriate value, or increase Air1 flow and/or increase the combustor flow to create a hotter flame in the combustor.
Problem with hydrogen gas supply.	Check the hydrogen gas supply as follows: • Is the hydrogen flow rate correct? • Is the hydrogen line plumbed correctly? • Is a hydrogen gas source connected to the hydrogen line? • Are the inlet manifold and tank connections free of leaks or other problems? • Is the hydrogen line obstructed (e.g., valve in the hydrogen line is turned off)?

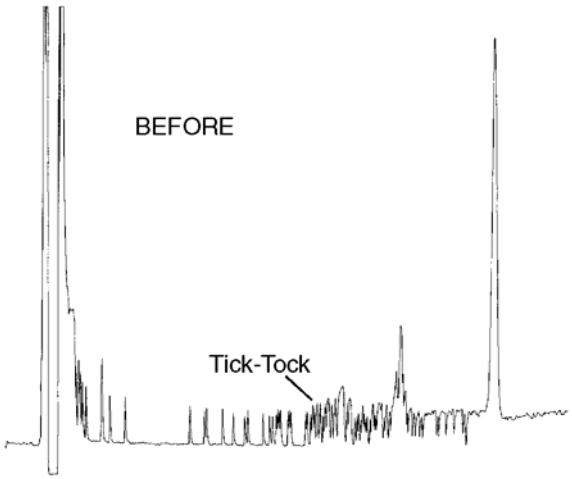
Baseline Noise is Too High

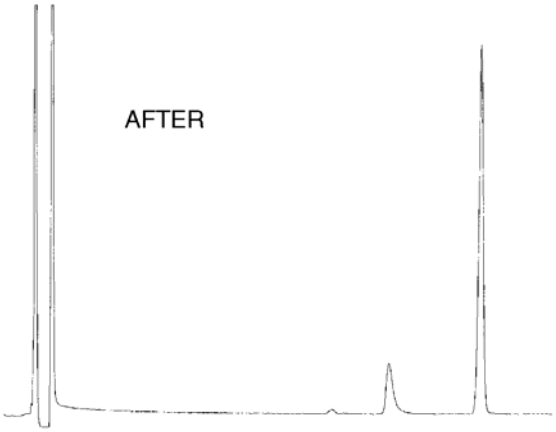
Possible Cause	*Solution*
Gas flows are improperly set.	Check the gas flow rates and readjust.
Combustor is dirty.	Bake the detector at high temperature overnight with flame pulsing. Replace the quartz combustor only if this treatment does not improve the baseline.
Column end is dirty or broken, or extends too far into the combustor.	The end of the column may be dirty, the column may have broken off in the combustor, or the column may be inserted too far into the combustor. If this is the case, splinters of glass may be fluorescing, thereby increasing the background signal. Reinstall the column.
Detector parts are dirty.	Detector parts may be contaminated from handling. Mild contamination often responds to a high temperature bakeout. If this proves unsuccessful, clean the parts with reagent grade iso-propanol in an ultrasonic bath. Repeat with several rinses of iso-propanol.
Light pipe window is dirty or broken.	Contact Varian Service.
Photomultiplier tube is aging.	As the PMT ages the voltage must be increased to achieve the same gain. This results in greater dark current noise. Replace the photomultiplier tube when acceptable detectivity can no longer be achieved.
Photomultiplier tube gain is too high.	Decrease the photomultiplier tube voltage to an acceptable level.
Gate Width/Gate Delay is incorrect.	The Gate Delay has been set too close to the hydrocarbon emission. Reset the Gate Delay.

Possible Cause	*Solution*
Normal/Square Root setting is incorrect.	Check that the Square Root Mode is not On, unless you wish to operate in that mode, in which case a high noise level is expected.
Gas flow rates are unstable.	Check for leaks. Check that the mass flow controllers are operating properly. Replace if necessary. **NOTE: To prevent steps in the baseline when the AutoSampler cycles, DO NOT drive the AutoSampler with the PFPD air line.**
Detector temperature is too high.	This is a particular problem when using the near-IR sensitive R5070 PMT at temperatures above 320 °C, as the PMT is sensitive to emissions from the walls of the detector itself. If you cannot operate the detector at a lower temperature, you may need to add a short pass filter which will block the near-IR wavelengths.
Detector is in the tick-tock mode.	Tick-tock mode indicates that the igniter volume is filling faster than the combustor volume. The cure is to either slow the igniter volume fill rate, or speed the combustor volume fill rate. To decrease the igniter volume fill rate, while viewing the baseline, slowly decrease the Air2 flow rate until the noise abruptly decreases. Then, turn the Air2 mass flow controller 1/10 of a turn more. To increase the combustor volume fill rate, turn the split valve counterclockwise slowly while watching the baseline. When the pulse becomes regular and background becomes constant, increase the split valve an additional 1/2 turn.
Trigger Level Set Incorrectly.	Increase or decrease trigger level ~20%. You may need to re-optimize Gate Delay after changing trigger level.
Flame propagating past end of the combustor support.	A rather characteristic pulse pattern (short pulse-long pulse-pause) suggests that the flame front is propagating past the end of the combustor support. This may happen at high detector temperatures or when using a narrow-bore column with hydrogen as the carrier gas. In such situations, the polyimide coating on the column end will be burned off below the combustor support. Increase the *Hydrogen* flow rate by ~10%. This cools the flame and limits the propagation of the flame below the end of the combustor support. Alternatively, you may connect your analytical column to a 15 cm length of 0.53 mm ID deactivated fused silica tubing using a Press-Fit connector, inserting the deactivated tubing into the PFPD in place of your analytical column. This minimizes the clearance between the column and the combustor, preventing unwanted flame propagation.

Baseline is Erratic (tick-tock)

Possible Cause	*Solution*
Ratio of combustor flow to igniter flow is too small.	If possible, increase the center flow rate by turning the split valve 1/4 turn counterclockwise. Decrease the Air2 flow rate.





DETECTOR TEST CHROMATOGRAMS

The following table lists the series of test samples available for Varian GC detectors.

Test Sample	*Part Number*	*Compound Concentrations*
TCD	82-005048-01	3.00 μg/μL of C_{14}, C_{15} and C_{16} in iso-octane
ECD	82-005048-02	33.0 pg/μL of lindane and aldrin in iso-octane
FPD	82-005048-03	20.0 ng/μL of n-dodecanethiol, tributylphosphate, methyl parathion; 4000 ng/μL of n-pentadecane in iso-octane
TSD	82-005048-04	2.00 ng/μL of azobenzene, methyl parathion; 4.00 ng/mL malathion and 4.00 μg/μL C_{17} in iso-octane
FID	82-005048-07	30 ng/μL of C_{14}, C_{15} and C_{16} in iso-octane
Note: If the FID sample is not available, the TCD test sample can be used if first diluted 100:1		

Table 61: Detector Test Samples

FID Test Mix

The following figures show typical chromatograms of the Varian test mixes for the detectors.

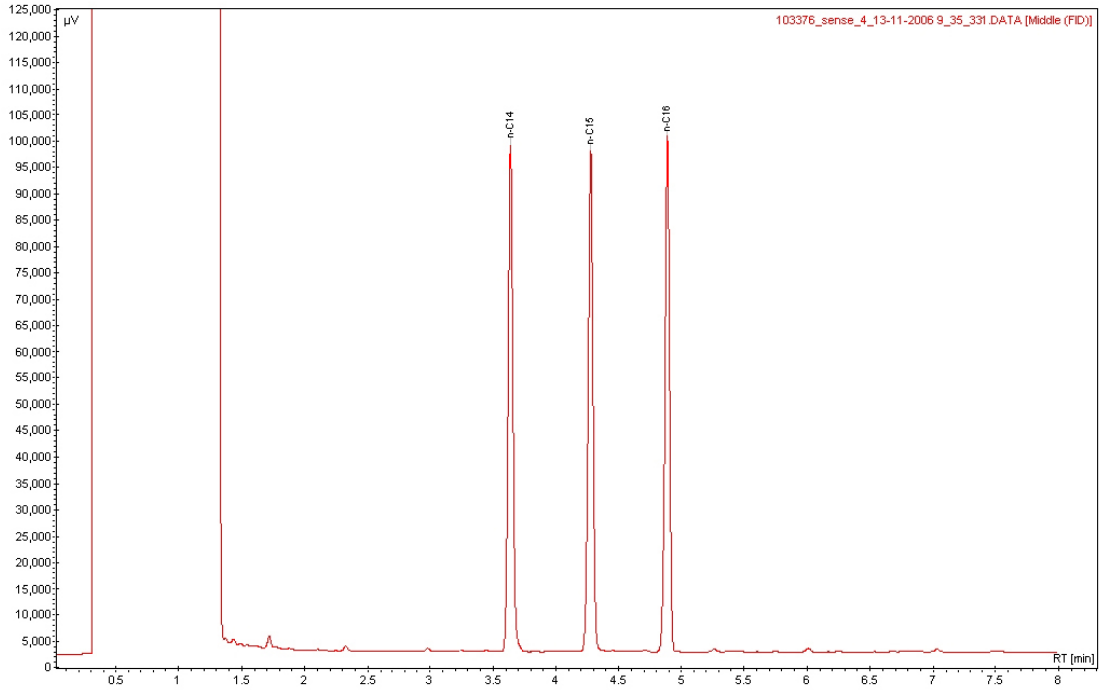


Figure 61: FID Test Mix Chromatogram

Column: Non-polar, e.g., 0.25µm film thickness, 15m x 0.25 mm, CP-Sil 5 CB
Carrier: Nitrogen, 5 mL/minute
Injection: 1079 PTV, 200 °C, 1 µL splitless
Temperature Program: 80 °, hold 1 minute, 20°/minute to 200°, hold 1 minute
Detector: 300 °C, Range 12

TSD Test Mix

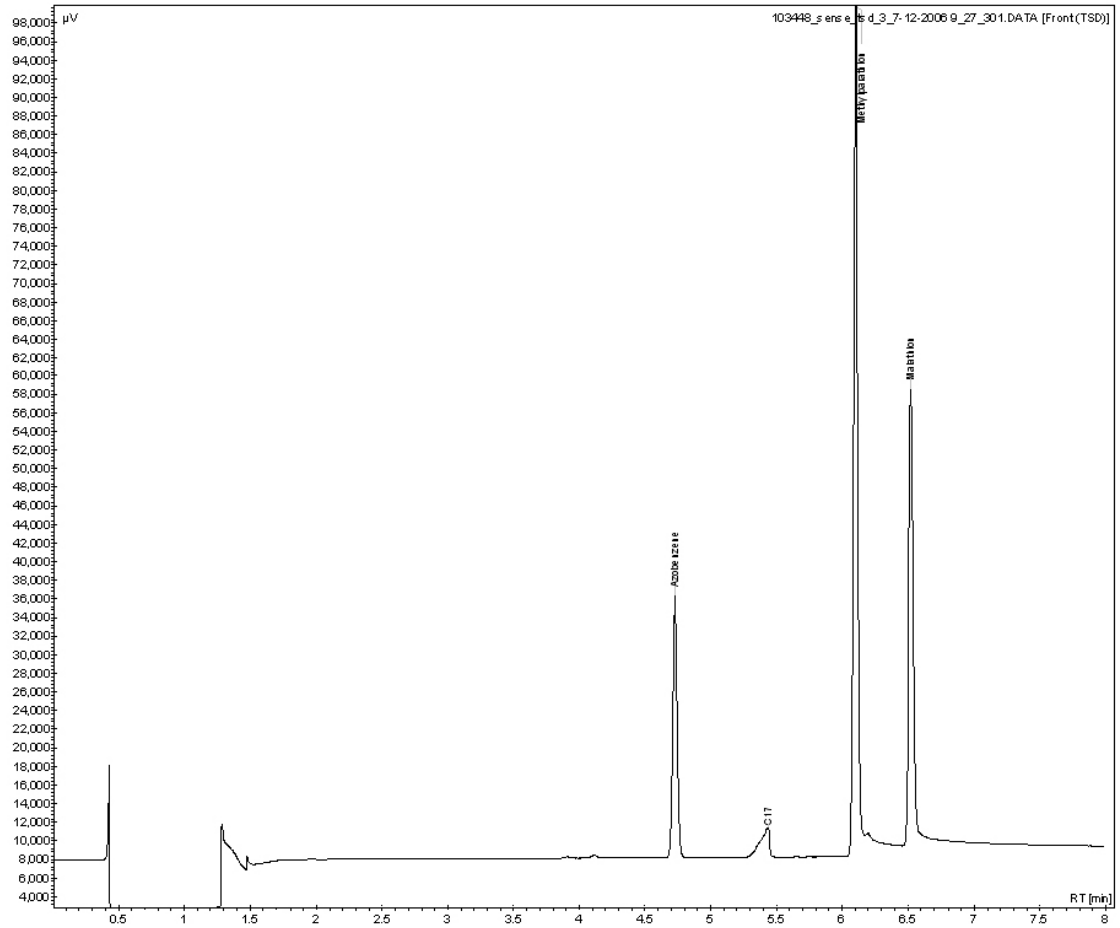


Figure 62: TSD Test Mix Chromatogram

Column: Non-polar, e.g., 0.25µm film thickness, 15m x 0.25 mm, CP-Sil 5 CB
Carrier: Nitrogen, 5 mL/minute
Injection: 1079 PTV, 200 °C, 1 µL splitless
Temperature Program: 80°, hold 1 minute, 20°/minute to 200°, hold 1 minute
Detector: 300 °C, Range 12

TCD Test Mix

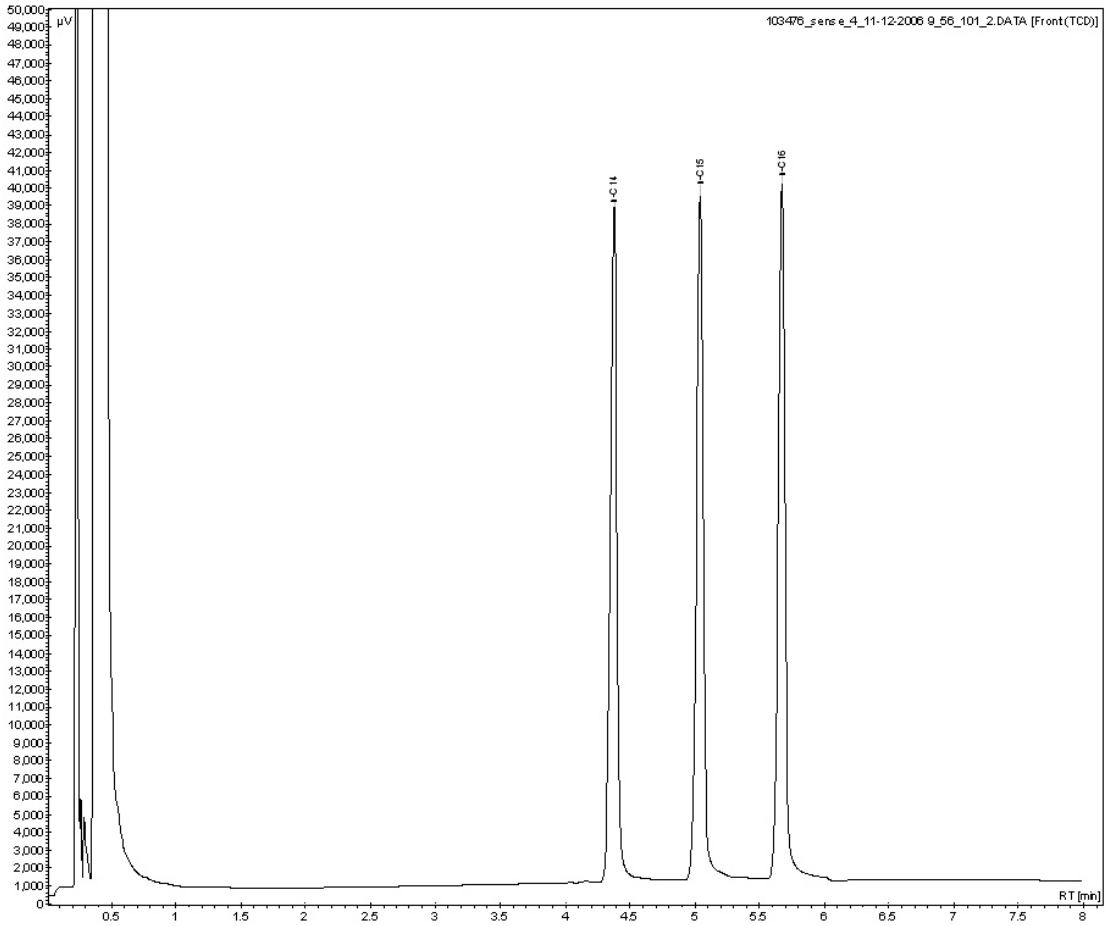


Figure 63: TCD Test Mix Chromatogram

Column: Non-polar, e.g., 0.25µm film thickness, 15m x 0.25 mm, CP-Sil 5 CB
Carrier: Helium, 5 mL/minute
Injection: 1079 PTV, 200 °C, 1 µL splitless
Temperature Program: 80°, hold 1 minute, 20°/minute to 200°, hold 1 minute
Detector: 220 °C, Range 0.05, Filament Temperature 270° ± 20°, Filament current 200 ±10 mA

ECD Test Mix

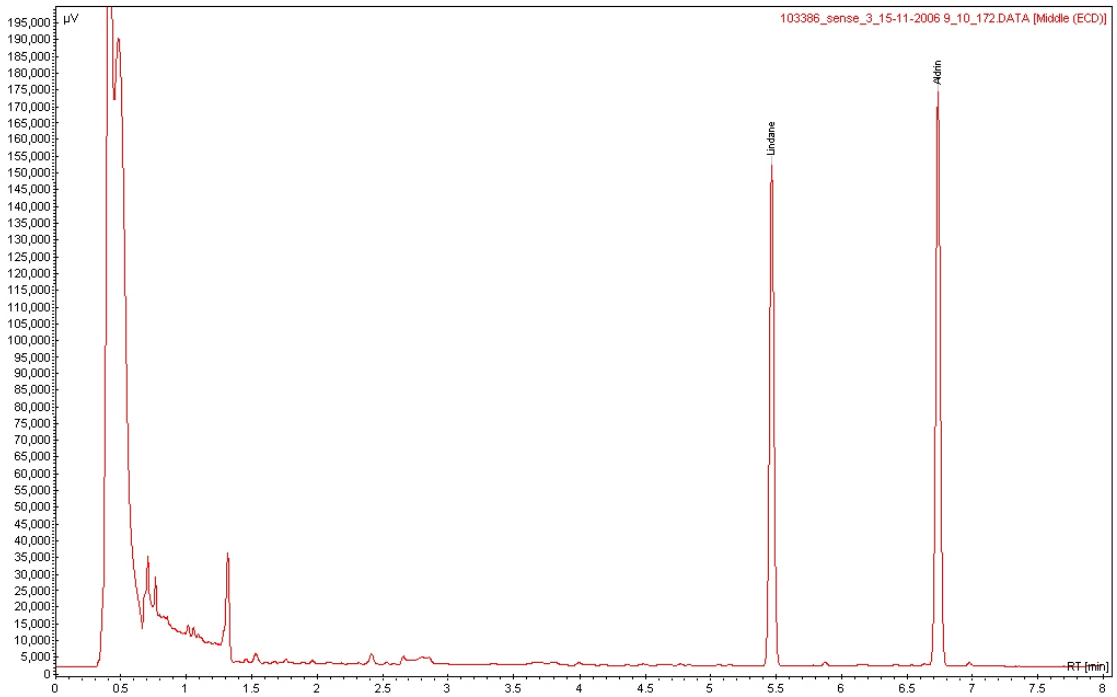


Figure 64: ECD Test Mix Chromatogram

Column: Non-polar, e.g., 0.25µm film thickness, 15m x 0.25 mm, CP-Sil 5 CB
Carrier: Nitrogen, 5 mL/minute
Injection: 1079 PTV, 200 °C, 1 µL splitless
Temperature Program: 80°, hold 1 minute, 20°/minute to 200°, hold 1 minute
Detector: 300 °C, Range 1, Capillary current setting

PFPD P Mode

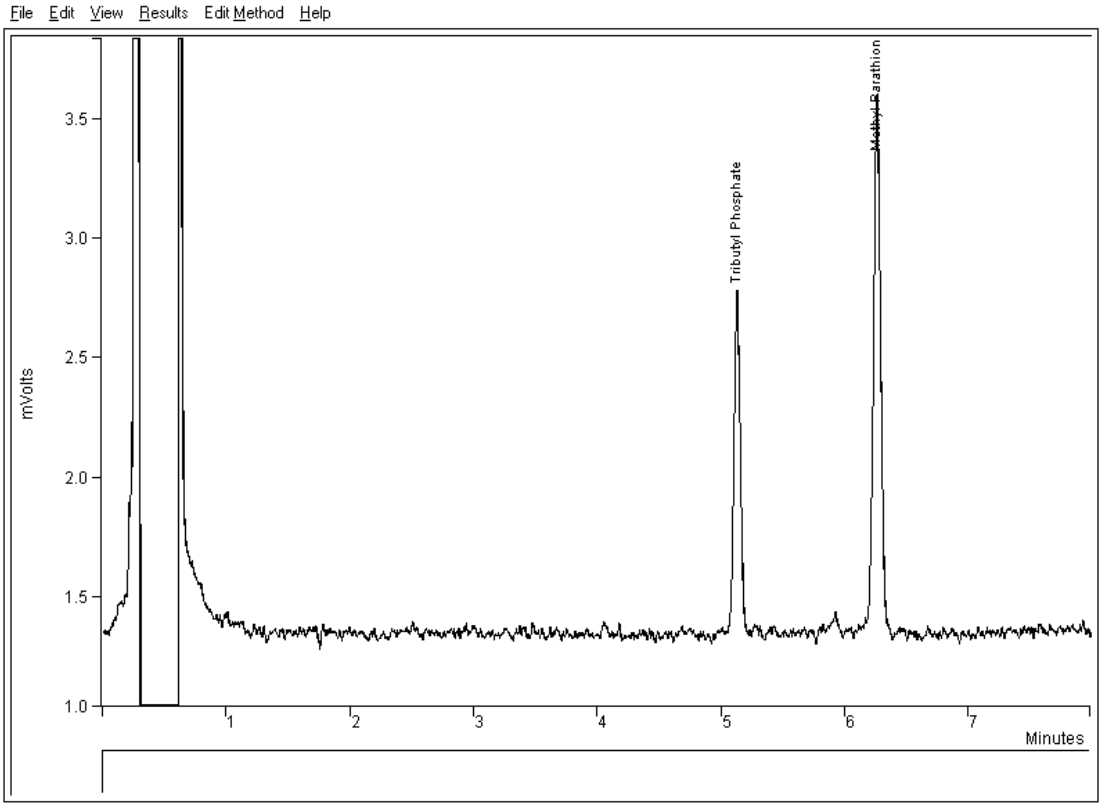


Figure 65: PFPD P Mode Test Mix Chromatogram

Column: Non-polar, e.g., 0.25µm film thickness, 15m x 0.25 mm, CP-Sil 5 CB
Carrier: Nitrogen, 2 mL/minute
Injection: 1079 PTV, 220 °C, 1 µL splitless of a 1:30 dilution of FPD test mix in iso-octane
Temperature Program: 80°, hold 1 minute, 20°/minute to 200°, hold 1 minute
Detector: 300 °C, Range 10, Air1 17 mL/min., Hydrogen 14 mL/min., Air2 10 mL/min.
Gate Delay 4 msec., Gate Width 10 msec, Trigger level 200 mV

PFPD S Mode

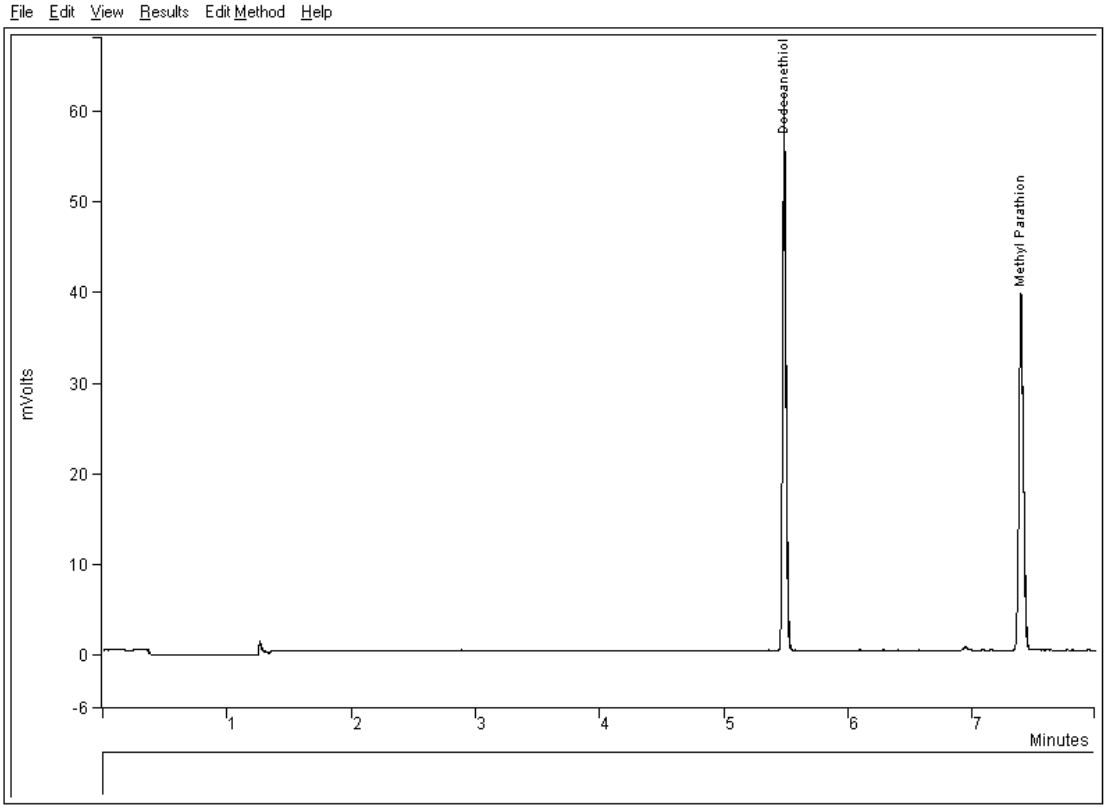


Figure 66: PFPD S Mode Test Mix Chromatogram

Column: Non-polar, e.g., 0.25µm film thickness, 15m x 0.25 mm, CP-Sil 5 CB
Carrier: Nitrogen, 2 mL/minute
Injection: 1079 PTV, 220 °C, 1 µL splitless of a 1:30 dilution of FPD test mix in iso-octane
Temperature Program: 80°, hold 1 minute, 20°/minute to 200°, hold 1 minute
Detector: 300 °C, Range 10, Air1 17 mL/min., Hydrogen 14 mL/min., Air2 10 mL/min.
Gate Delay 6 msec., Gate Width 20 msec, Trigger level 200 mV

GENERAL GC MAINTENANCE

Overview

In this section maintenance information is presented in 4 headings:

1. General GC maintenance, on page 428.
2. Injector maintenance, on page 432.
3. Detector maintenance, on page 436.
4. Pencil Filters, on page 437,

Note that some maintenance procedures, such as repair or replacement of electronic components, should be performed by a qualified Varian service representative. For maintenance problems where mechanical or electrical assemblies need to be repaired or replaced please call your local Varian service center.

There are certain maintenance tasks that should be performed on a routine basis. These regular procedures are done to ensure that the 450-GC will continue to operate at optimum performance. The following is a brief summary of some common maintenance tasks and their typical frequency:

Maintenance Task	***Frequency***
Change septa	Typically 50 - 100 injections
Check column nuts are tight	Daily, or when performance determinate
Condition column	Daily or as required
Change Injector inserts	Weekly, or as required
Check gas supplies	Weekly
Leak check	Monthly
Change gas cylinders	Quarterly or as required
Condition system	Semiannually
Clean detectors or parts	When performance deteriorates
Replace gas purifiers	Annually or as required
Pencil Filters	On a yearly base.
Change/Replace column	When performance deteriorates

Table 62: Maintenance Frequency

General GC Maintenance

The common general GC maintenance tasks are checking and changing gas supply cylinders, leak checking and changing gas purifier filters. These should be performed at the frequency suggested above. The following procedures are critical to the successful long term operation of a Gas Chromatograph. It is very important to leak check the system on a routine basis and to ensure the quality of gas supplies, particularly the carrier gas and H_2 (Safety Reasons).

Check and Renew Gas Supplies

The pressure of the various GC gas supplies should be checked on a weekly basis and the following guidelines used for frequency of renewing supplies:

Carrier gas: The carrier gas supply cylinder should be changed when its pressure drops below 200 psi. This ensures that high purity carrier gas is always supplied to the instrument. With typical usage on one Gas Chromatograph an A-size cylinder of carrier gas should last for 3 to 6 months. Note that this usage includes also using the carrier gas as the supply of make-up to the detectors. When a new cylinder is installed the regulator and tubing should be purged with carrier gas before connecting to the GC. This will avoid introducing a large amount of air into the GC. Flush the GC for a few minutes before heating the Column.

Detector fuel gases: The typical detector fuel gases are hydrogen and air. These should be changed when the supply pressure drops below 200 psi. Typically an A size cylinder of hydrogen used on one GC with one Flame Ionization Detector lasts about 6 months. However, due to the high flow rate of air used in a Flame Ionization Detector this supply will need renewing more frequently. For this reason it is worthwhile to consider a clean supply of compressed air for FID equipped instruments.

Leak Check

Leak checking is one of the most important maintenance tasks for any Gas Chromatograph. The following information refers to general leak checking procedures for the 450-GC. Specific information regarding individual 450-GC components will be presented in the relevant part of this maintenance section.

Leaks in the GC system can lead to poor chromatographic performance or damage components such as the analytical column. The presence of oxygen in the GC carrier gas at elevated temperatures can lead to permanent column phase degradation. While the use of an oxygen filter on the carrier supply to the instrument can help, leaks downstream of the filter are generally more likely to be the problem.

The use of soap-based leak detection fluids is ***NOT*** recommended for a high performance capillary Gas Chromatograph due to the danger of introducing contaminants into the system. If leak detection fluids are being used they should only be used outside the Gas Chromatograph, i.e. to verify there are no leaks in the plumbing to the Gas Chromatograph. If a leak detection fluid is being used inside the Gas Chromatograph, an alcohol such as isopropanol or a 50:50 mixture of isopropanol and water should be used. Use a dropper or syringe to place a few drops of leak detection fluid on the desired fitting and monitor the area for bubble formation.

The most important step in leak checking is to verify that the GC system can hold pressure. This is done by removing the column from the Injector, sealing all exits from the Injector and pressurizing the system. Use the following procedure to leak check 450-GC Injectors:

When conducting leak check procedures it is important to completely seal all carrier gas exits, including septum purge and split vent outlets. If more than one Injector is present, then the outlets of all the Injectors must be sealed. The following procedure refers to one Injector but should be duplicated for every Injector on the 450-GC.

1. Set the column oven and the Injector zone temperatures to 50°C and allow them to reach this temperature. This allows safe handling of Injector surfaces.

2. Remove Injector septum nut and install a new septum. An older septum can often be the source of a substantial leak.

3. Remove the column from the Injector. Use the appropriate nut and ferrules to seal the base of the Injector. For packed column systems use a 1/4" Swagelok blank off plug (SWB400P). For capillary systems use a capillary nut (03-949551-00) with a no hole ferrule (28-694590-01).

4. If the Injector is either an 1177, 1093 or a 1079 PTV, then the septum purge and split vent outlets must be sealed. The septum purge outlets are located on the top frame surrounding the column oven, behind the column oven door. The split vent outlets are located on the left side panel of the 450-GC. Seal the septum purge by removing the outlet fitting from the septum purge valve and replacing it with a blank off plug (P/N SWB200P). Seal the split vent outlet by installing a Swagelok union on the outlet tube and sealing it with a blank off plug.

5. With all outlet ports plugged, pressurize the system to 20 - 30 psi. This can be accomplished by adjustment of manual pneumatic controls or Electronic Flow Control (make certain that the split ratio is turned to off, constant flow is not selected and the septum purge valve, if appropriate, is turned off). For information on setting carrier gas pressure see the Basic Operation section of this manual.

6. Shut off the carrier supply at the source and monitor the displayed pressure at the GC for 15 minutes. The pressure should not drop more than 0.5 psi in 15 minutes.

Leak checking ensures that there are no leaks in the GC system up to and including the Injector assembly. If the indicated pressure on the GC drops by more than 0.5 psi (3 kPa) during the 15 minute test period, this indicates that there is a positive leak in the system. Finding such a leak, particularly if it is a small leak, can be very difficult. In general the best approach is to systematically go through the pneumatic system from the source and tighten each fitting until the leak is eliminated. It is important to note that leaks are often found in the carrier gas supply to the GC.

Locate leaks preferably with an electronic leak detector or leak detection fluid. An electronic helium leak detector is available from Varian (CP87610 110 VAC, CP80610 220 VAC, CP83610 240VAC). This device can detect helium concentration in the air as low as 2 ppm and is very effective at identifying the precise location of a helium leak from a GC system.

Gas Purifier Replacement

The most common gas purifiers used in gas chromatography are moisture traps for removing water vapor, hydrocarbon traps for removing low levels of hydrocarbon contaminants and oxygen traps for removing low levels of oxygen. In general the choice of filter depends on the application and detector used. Carrier gas should contain less than 1 ppm oxygen, moisture and trace hydrocarbon contaminants. A moisture and a hydrocarbon filter are recommended for general applications to maintain detector background levels at their lowest possible level. If the detector used is an ECD or MS an oxygen filter should be employed as well.

Carrier gas filters are typically located on the side or at the rear of the 450-GC. Varian supplies several types of carrier gas purifiers. These include a hydrocarbon trap, moisture trap and oxygen trap. While the choice of purifier depends on the individual configuration and performance expectations, the following configuration is recommended for maximum carrier gas purity and protection.

The carrier gas should first pass through a hydrocarbon trap, then a moisture filter and then an oxygen trap. As moisture reduces the effectiveness of an oxygen trap, a moisture trap should always be placed in front of an oxygen trap. The moisture trap should also be changed immediately when it changes color to prevent breakthrough of moisture to the oxygen trap.

The minimum recommended carrier gas purifier configuration is a moisture and hydrocarbon trap. This configuration can be used with routine GC analyses using FID, TCD, TSD and PFPD detectors. If an ECD or mass spectrometer is being used, an oxygen trap should also be added. For trace analysis the combination of hydrocarbon trap, moisture trap and oxygen traps should be used.

To install or replace the Gas Clean filter cartridges: remove new filter from sealed bag. Unscrew the ring on the filter base, lift up the filter cartridge. Always replace the 2 upper O-rings. The base contains check valves that shut off flow when the cartridge is removed. Place ring over new cartridge, press and hold cartridge to base and engage retaining ring.

The following are the gas purifiers available from Varian.
Check the Varian website for more information: www.varianinc.com.

Varian Gas Clean Filters	***Part Number***
Gas Clean oxygen filter	CP17970
Gas Clean charcoal filter	CP17972
Gas Clean moisture filter	CP17971
Gas Clean GC/MS filter	CP17973

Table 63: Varian Gas Clean Filters

Injector Maintenance

The Injector is the component of the Gas Chromatograph that requires the most frequent maintenance. This is due to the fact that the sample is deposited in the Injector, thus leading to potential contamination and build up of non volatile deposits. The most frequent Injector maintenance is septum replacement. In addition insert replacement and Injector cleaning are very common. As septum replacement is common to all liquid Injectors on the 450-GC this procedure is presented first, followed by instructions for High-Temperature bake out of the Injector, followed by specific maintenance procedures for the individual Injectors.

Septum Replacement

The Injector septum is an expendable part and must be replaced on a routine basis. The frequency of replacement depends on the number of injections and whether the injections are by hand or with an autosampler. In general the septum should be replaced every 50 - 100 injections or when symptoms of a septum leak are seen. These symptoms include changing retention times, reduced detector response or a drop in column head pressure. The latter symptom is not always valid as some Injectors, such as the 1079 PTV, are pressure controlled. With a pressure controlled Injector, the column head pressure will remain constant even if a leak is present.

Use the following procedure to change a septum on any Varian Injector:

1. Cool the column oven and Injector oven to 50°C. This ensures safe handling of Injector parts and protects the column from elevated temperatures while it is exposed to air.

2. Turn off the carrier gas supply to the Injector to prevent the septum from being inadvertently ejected due to carrier gas pressure.

3. Remove the Injector nut by turning it counterclockwise.

4. Remove the old septum using metal tweezers or a septum pick (P/N 72-000084-00). Do not to handle any internal parts of the Injector with hands or gloves.

5. Install the new septum, again using tweezers to avoid contaminating the Injector.

6. Replace the Injector nut and tighten it finger-tight until you feel resistance from the septum, then tighten an extra 1/4 turn. Turn on the carrier gas supply.

On occasion to save time you may want to change a septum while the Injector is hot. Use an Injector wrench (P/N 03-908423-00) to remove and reinstall the Injector nut. In all instances the column oven should be cool before removing the Injector nut.

Instructions for High-Temperature bakeout of Injectors

From time to time, low volatility contaminants may build up in the Injector to a level which interferes with proper operation. One way to remove these contaminants is by a high temperature bakeout. Temperature sensitive parts are removed from the Injector and replaced with the Bakeout nuts and seals described below. This procedure applies to 1177 and 1079 PTV Injectors installed on Varian 450-GC or 430-GC.

Injector	*Item*	*Part Number*	*Description*
1177	Bakeout NUT	03-925217-01	BLACK
	Bakeout DISK	03-925731-02	0.34"OD x 3/32"
1079 PTV	Bakeout NUT	03-925772-01	RED
	Bakeout DISK	03-925216-02	0.55"OD x 3/32"

Table 64: Injector Bakeout Parts

1. Verify the GC is capable of normal operation and that clean, inert carrier gas is supplied. Handle all parts using clean tweezers and place all parts to be re-used on clean lab tissues. *Remember that it is much easier to*

maintain cleanliness than it is to remove contaminants once they have been introduced into the system.

2. Uninstall the analytical column from the Injector(s) to be baked out and install a minimal flow restriction column such as 03-912805-99, or any piece of scrap column more than 5 meters long and less than 0.53 mm ID, using a graphite ferrule such as SG072627. The restrictor column should be installed so that it just protrudes beyond the end of the ferrule into the Injector, to allow the purge gas to sweep out the entire Injector volume down to the ferrule. **Do not install the scrap column into the detector!** Insert the free ends of the analytical column into a clean septum to prevent the entry of contaminants during the bakeout period.

3. Enter Setup and edit the column parameters to reflect the restrictor column you installed. Change the Gas Saver Timeout to 0.00 minutes. Change the temperature limit(s) for the Injector(s) to be baked out to at least 400°C.

4. Remove and discard the septum from the Injector(s). Take care to remove any septum residues which may adhere to Injector surfaces to avoid further contamination of the Injector with septum decomposition products during bakeout. Remove the Injector insert from the Injector(s) and replace with a clean insert and graphite O-ring (03-926119-40 1177 Injector). Note that any deactivation treatment on the insert will probably be destroyed during bakeout, so you may wish to retain this insert for use in future bakeouts.

5. Install the appropriate bakeout disk in the Injector and finger tighten the special bakeout Injector nut. Set up the carrier gas flow to give a column flow of about 5 mL/minute, a septum purge flow of about 10 mL/minutes and a split ratio of 20 or more. Be sure that the Injector is set up in Split mode. Slowly tighten the Injector nut until the Injector vent flow reaches the expected value for the column flow and split ratio. When this value is reached, the Injector is approximately sealed. A wrench may be required to apply sufficient torque to make an adequate seal, but do not overtighten to the point that the Injector is damaged. It is not necessary that the Injector be sealed leak tight.

6. Set the Injector temperature to 400 °C and bake out for about 4 hours. During this time you may want to maintain a column oven temperature of about 300 °C to assist in cleaning the Injector crossover. However, do not heat the column oven above the temperature limit of any components, such as valves, etc. which will remain in the oven during bakeout.

7. Cool the Injector and column oven to about 50 °C. Remove the bakeout nut, bakeout seal, insert and sealing ferrule and scrap column and replace with components appropriate to the intended analysis. Reset the column parameters, Gas Saver Timeout and temperature limits you changed in Setup. Restore the desired GC method parameters.

8. To minimize contamination in the future, you may wish to install Varian's new Bleed and Temperature Optimized septa, see the specific Injector section for more details about recommended septa.

Injector specified maintenance

1177 Injector, click here, on page 171.

1079 PTV Injector, click here, on page 195.

1041 Injector, click here, on page 215.

1061 Injector, click here, on page 215.

1093 Injector, click here, on page 245.

Detector specified maintenance

Flame Ionization Detector (FID), on page 292.

PDHID Detector, on page 296.

Thermionic Specific Detector (TSD), on page 317.

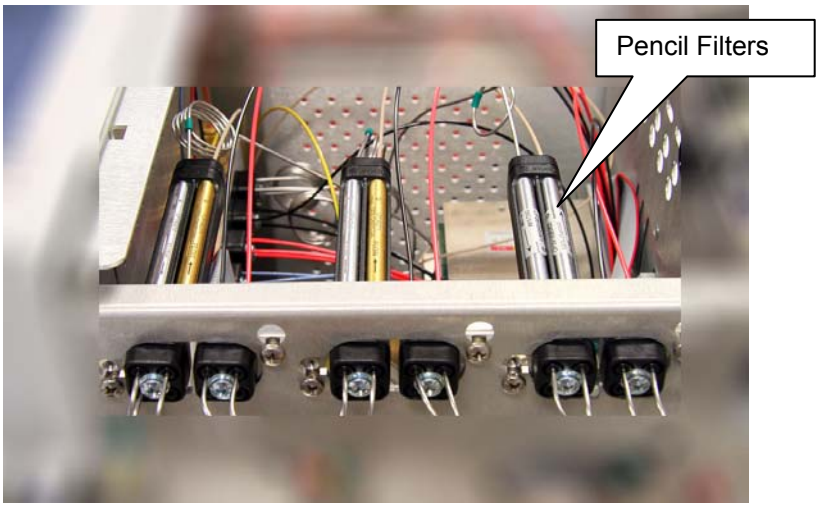
Thermal Conductivity Detector (TCD), on page 328.

Electron Capture Detector (ECD), on page 340.

Pulsed Flame Photometric Detector (PFPD), on page 409.

Pencil Filter

The Pencil filters are used to protect the Injector and/or EFC (Injector depending).
The Pencil Filter(s) are located underneath the left-Top cover.
Both pencil filters must be replaced on a yearly base.
It is advised to have the filters replaced by a Varian service engineer on a regular maintenance visit.



2 types of Pencil Filters are available:

1. Molsieve/Charcoal filter (CP742210), silver color.

2. Charcoal filter (CP742211), gold color.

SHIPPING, CLEANING AND DISPOSAL INSTRUCTIONS

SHIPPING INSTRUCTIONS

CAUTION

If your 450-GC for any reason must send back to the nearest Varian, Inc. subsidiary or Varian, Inc. representative, it is very important to follow these additional preparation instructions:

1. Place the shipping restrain pin back.
2. Put protection plugs on the sample and/or carrier gas inlets.
3. Remove columns.

CLEANING INSTRUCTIONS

To keep the 450-GC surface clean, refer to the remarks given below:

- Switch the 450-GC off.
- Remove the power cable.
- Put protection plugs on the sample and carrier gas inlets.
- Put protection plugs on the Column vents.
- Use a soft (no hard or abrasive) brush to carefully brush away all dust and dirt.
- If the outer case is dirty (never clean the inside!) clean it with a soft, clean cloth dampened with mild detergent.
- Never use alcohol or thinners to clean the 450-GC, these chemicals can damage the case.
- Be careful not to get water on the electronic components.
- Do not use compressed air to clean.

DISPOSAL INSTRUCTIONS

When the lifetime of the 450-GC or parts of it has reached the end of its useful life, disposal must be carried out in accordance with all (environment) regulations applicable in your country.

REPLACEMENT PARTS

Replacement Parts for the CP-8400 AutoSampler, CP-8410 AutoInjector, 1177, 1041, 1061, 1079 PTV and 1093 Injectors, FID, TSD, ECD, TCD and PFPD detectors.

CP-8400/8410 Vials			
2 mL Vials	2 mL Ultra GC/MS vials, screw top wide opening clear glass, 9 mm caps, w/ Ultra GC/MS septa	392611979	100/pk
	2 mL Ultra GC/MS vials, screw top wide opening amber glass, 9 mm caps, w/ Ultra GC/MS septa	392612016	100/pk
	2 mL screw top wide opening clear glass vials, 9 mm caps w/ PTFE/Silicone septa	392611549	100/pk
	2 mL screw top wide opening amber glass vials, 9 mm caps w/ PTFE/Silicone septa	392611550	100/pk
	2 mL crimp top wide opening clear glass vials, 11 mm caps w/ PTFE/rubber septa	392611518	100/pk
	100µl polypropylene crimp top vial; 12 X 32 mm, 11mm caps with PTFE/Butyl rubber septa	392611669	100/pk
	Crimping tool, 11 mm	AL666011	1 pc
5 mL Vials	5 mL Ultra GC/MS vials, 20 mm x 38 mm solvent vials and CP-8410 sample vials w/ snap cap and Ultra GC/MS septa	392611980 392611981 392611982	4/pk 50/pk 100/pk
	5 mL 20 mm x 38 mm solvent vials and CP-8410 sample vials w/ snap cap and septa	392611970 392611975 392611974	4/pk 50/pk 100/pk
10 mL Vials	10 mL Ultra GC/MS vials, 22 mm x 45 mm sample vials for CP-8410 w/ snap cap and Ultra GC/MS septa	392612006 392612007	50/pk 100/pk
	10 mL 22 mm x 45 mm sample vials for CP-8410 w/ snap cap and septa	392611973 392611972	50/pk 100/pk
Starter Kit	Ultra GC/MS Starter Kit (10 ea. 2 mL vials with caps and Ultra septa, 6 ea. 5 mL vials and 5 ea. 10 mL vials with 11 wide hole snap caps with Ultra septa)	392611978	1 ea

CP-8400/8410 Syringes			
1 µL	Hamilton 1 µL 7701 N Syringe	HM202950	1 ea
2 µL	Hamilton 2 µL 7702 N Syringe	HM202960	1 ea
5 µL	Hamilton 5 µL 701 syringe, 26 gauge	HM87900	1 ea
	SGE 5 µL syringe, conical tip needle, 26 gauge	392590204	1 ea
10 µL	Hamilton 10 µL 701N syringe, 26 gauge	HM80300 HM80366	1 ea 6/pk
	Hamilton 10 µL 701 syringe, conical tip needle, 26 ga.	SK0163520	1 ea
	SGE 10 µL syringe, conical tip needle, 26 gauge	SG002980	1 ea
100 µL	SGE 100 µL syringe, Teflon tip, removable needle	SG005333	1 ea
250 µL	SGE 250 µL syringe	SGS006157	1 ea

CP-8400/8410 Replacement Carrousel/Tray			
	CP-8400 AutoSampler 100-vial Sample Carrousel	CP738642	1 ea
	CP-8410 AutoInjector Sample Tray	CP739656	1 ea

Quick Connect Ferrules and Column Scales			
	Graphite Quick Connect ferrules w/ brass jackets (not for use connecting to MS detector)		
	0.4 mm, for 0.25 mm ID columns	392538401	2/pk
	0.5 mm, for 0.32 mm ID columns	392538402	2/pk
	0.8 mm, for 0.53 mm ID columns	392538403	2/pk
	450/430 Column insertion scale	392575001	1 ea

SPME Parts			
Fiber	SPME Fiber assortment 3/pk, 24ga	391896308	3/pk
Test Sample	SPME test sample, pk 5 ampules	391896700	5 ea
Fiber Holder	SPME fiber holder, CombiPAL/CP-8400/CP8410	392590210	1 ea

Injector Liners/Inserts

Insert Type[a]	Description	1041	1061	1079 PTV	1093	1177
1	Split liner with fused silica wool					
2	Laminar cup split liner					
3	Split liner with frit					
4	Baffle split liner					
5	Splitless liner					
6	Liner for direct injection on narrow bore columns, SPI			X	X	
7	Liner for on-column 0.53 mm ID columns, SPI			X	X	
8	SPI Glass liner with buffer				X	
9	On-column injection liner	X				
10	Splitless liner					X
11	Splitless liner					X
12	Gooseneck Split/Splitless liner					X
13	Gooseneck Split/Splitless liner					X
14	Gooseneck Split/Splitless with glass wool					X
15	Double Gooseneck Splitless liner					X
16	Inverted Cup liner.					X
17	Inverted cup with fused silica wool					X
18	Inverted cup liner packed with 10% OV-1 on Chromosorb WHP					X
19	Straight liner with quartz wool					X
20	Gooseneck Split/Splitness liner					X
21	Straight Split/Splitless liner with glass wool					X
22	Splitless liner with glass wool					X

[a] Please refer to Liner/Insert Description on page 445

23	Split liner packed with 10% OV-1 on Chromosorb WHP					
Insert Type[a]	**Description**	**1041**	**1061**	**1079 PTV**	**1093**	**1177**
24	Split/Splitless Liner					
25	Liner for direct injection on Megabore 0,53 mm		X			
26	Splitter insert with glass wool		X			
27	Gooseneck splitter insert			X		
28	Gooseneck splitter liner with frit			X		
29	Splitless liner			X		
30	Liner for SPME			X		
31	Splitless insert			X		
32	SPI glass liner with packed glass wool				X	
33	Glass column guide for 3400, 3500, 3600					
34	Glass column guide for 3700, 450-GC					
35	Split/Splitless liner with glass wool			X		
36	Split liner packed with 10% OV-101 on Chromosorb WHP 80/100			X		
37	Splitless liner					X
38	Split/Splitless Liner					X
39	Split/splitless insert with gooseneck					X
40	Split insert with fused silica wool					X
41	Split/Splitless Fast Focusliner					
42	Split insert with Quartz wool					
43	Split/Splitless Focusliner					
44	Split/Splitless Focusliner					
45	Split/SplitlessTapered FocusLiner with restriction					
46	Straight- through Liner			X		

[a] Please refer to Liner/Insert Description on page 445

47	Split/Splitless Tapered FocusLiner			X		
Insert Type[a]	**Description**	**1041**	**1061**	**1079 PTV**	**1093**	**1177**
48	Split/Splitless Focusliner			X		
49	Split/Splitless Liner			X		
50	Splitless Liner with Taper			X		
51	Splitless Liner with slots					
52	Packed Liner for Large-Volume Injection (LVI)			X		

Liner/Insert Descriptions

Insert Type	Part Number	Description	Type	ID	OD	Length	Volume	Material	Wool_Material	Deactivated
1	SG092021	Split liner with fused silica wool	Gooseneck	4 mm	6.3 mm	72 mm	1000 µL	Glass	Quartz	Yes
2	190010902	Laminar cup split liner	Inverted cup	4 mm	6.3 mm	72 mm	1000 µL	Glass		yes
3	190010903	Split liner with frit	Gooseneck	4 mm	6.3 mm	72 mm	1000 µL	Glass		Yes
4	190010904	Baffle split liner	Baffle	4 mm	6.3 mm	72 mm	1000 µL	Glass		Yes
5	190010905	Splitless liner	Gooseneck	2 mm	6.3 mm	72 mm	250 µL	Glass		Yes
6	190010906	Liner for direct injection on narrow bore columns, SPI	Tapered	0.5 mm	5 mm	54 mm	10 µL	Glass		Yes
7	190010907	Liner for on-column 0.53 mm ID columns, SPI	Tapered	0.8 mm	5 mm	54 mm	30 µL	Glass		Yes
8	190010908	SPI Glass liner with buffer	Tapered	0.8 mm	4.6 mm	54 mm	30 µL	Glass		Yes
9	391720300	On-column injection liner	On-column					Metal		
10	392611924	Splitless liner	Straight	2 mm	6.5 mm	78.5 mm	0.25 mL	Glass		Yes
11	392611925	Splitless liner	Straight	4 mm	6.5 mm	78.5 mm	1000 µL	Glass		Yes
12	392611926	Gooseneck Split/Splitless liner	Gooseneck	2 mm	6.5 mm	78.5 mm	250 µL	Glass		Yes
13	392611927	Gooseneck Split/Splitless Liner	Gooseneck	4 mm	6.5 mm	78.5 mm	1000 µL	Glass		Yes
14	392611928	Gooseneck Split/Splitless with glass wool	Gooseneck	4 mm	6.5 mm	78.5 mm	1000 µL	Glass	Glass	Yes

[a] Please refer to Liner/Insert Description on page 445

15	392611929	Double Gooseneck Splitless liner	Gooseneck	4 mm	6.5 mm	78.5 mm	1000 µL	Glass		Yes
Insert Type	**Part Number**	**Description**	**Type**	**ID**	**OD**	**Length**	**Volume**	**Material**	**Wool_Material**	**Deactivated**
16	392611931	Inverted Cup liner.	Inverted Cup	4 mm	6.3 mm	78.5 mm	1000 µL	Glass		Yes
17	392611932	Inverted cup with fused silica wool	Inverted Cup	4 mm	6.3 mm	78.5 mm	1000 µL	Glass	Fused Silica	Yes
18	392611933	Inverted cup liner packed with 10% OV-1 on Chromosorb WHP	Inverted cup	4 mm	6.3 mm	78.5 mm	1000 µL	Glass		Yes
19	392611934	Straight liner with quartz wool	Straight	4 mm	6.3 mm	78.5 mm	1000 µL	Glass	Quartz	yes
20	392611936	Gooseneck Split/Splitness liner	Gooseneck	4 mm	6.5 mm	78.5 mm	1000 µL	Glass	Glass	Yes
21	392611937	Straight Split/Splitless liner with glass wool	Straight	4 mm	6.4 mm	78.5 mm	1000 µL	Glass	Glass	yes
22	392611938	Splitless liner with glass wool	Straight	2 mm	6.5 mm	78.5 mm	250 µL	Glass	Glass	Yes
23	392611941	Split liner packed with 10% OV-1 on Chromosorb WHP	Gooseneck	4 mm	6.3 mm	72 mm	1000 µL	Glass		Yes
24	392611942	Split/Splitless Liner	Gooseneck	1 mm	6.3 mm	72 mm	225 µL	Glass		Yes
25	392611943	Liner for direct injection on Megabore 0,53 mm	Direct injection				1000 µL	Glass		
26	392611944	Splitter insert with glass wool	Straight				1000 µL	Glass	Glass	Yes
27	392611945	Gooseneck splitter insert	Gooseneck	3.4 mm	5 mm	54 mm	500 µL	Glass		Yes
28	392611946	Gooseneck splitter liner with frit	Gooseneck	3.4 mm	5 mm	54 mm	500 µL	Glass		Yes
29	392611947	Splitless liner	Gooseneck	2 mm	5 mm	54 mm	250 µL	Glass		Yes
30	392611948	Liner for SPME	Straight	0.8 mm	5 mm	54 mm	30 µL	Glass		Yes
31	392611949	Splitless insert	Straight	0.5 mm	5 mm	54 mm	15 µL	Glass		Yes
32	392611950	SPI glass liner with packed glass wool	Tapered	0.8 mm	4.6 mm	54 mm	30 µL	Glass		Yes
33	392611951	Glass column guide for 3400, 3500, 3600			6 mm	63.5 mm		Glass		
34	392611952	Glass column guide for 3700, 450-GC		0.8 mm	4.6 mm	18.3 mm		Glass		
35	392611953	Split/Splitless liner with glass wool	Straight	2 mm	5 mm	54 mm	250 µL	Glass	Glass	Yes
36	392611954	Split liner packed with 10% OV-101 on Chromosorb WHP 80/100	Straight		5 mm	54 mm		Glass		Yes
37	RT207132145 5 pack	Splitless liner	Straight	2 mm	6.2 mm	78.5 mm	250 µL	Glass		Siltek
38	RT207732145 5 pack	Split/Splitless Liner	Straight	4 mm	6.5 mm	78.5 mm	1000 µL	Glass		Siltek

39	RT207992145	Split/splitless insert with gooseneck	Gooseneck	4 mm	6.2 mm	78.5 mm	1000 µL	Glass		Siltek
Insert Type	**Part Number**	**Description**	**Type**	**ID**	**OD**	**Length**	**Volume**	**Material**	**Wool Material**	**Deactivated**
40	RT21079 PTV2265	Split insert with fused silica wool	Straight	4 mm	6.5 mm	78.5 mm	1000 µL	Glass	Quartz	Siltek
41	SG092113	Split/Splitless Fast Focusliner	FocusLiner	2.3 mm	6.3 mm	72 mm	300 µL	Glass		Yes
42	SG092021	Split insert with Quartz wool	Gooseneck	4 mm	6.3 mm	72 mm	1000 µL	Glass	Quartz	Yes
43	SG092022	Split/Splitless Focusliner	Focusliner	4 mm	6.3 mm	72 mm	1000 µL	Glass	Quartz	Yes
44	SG092025	Split/Splitless Focusliner	Focus liner	4 mm	6.3 mm	72 mm	1000 µL	Glass	Quartz	Yes
45	SG092028	Split/SplitlessTapered FocusLiner with restriction	Focus liner	4 mm	6.3 mm	72 mm	1000 µL	Glass	Quartz	Yes
46	SG092031	Straight- through Liner	Straight	0.5 mm	5 mm	54 mm	10 µL	Glass		Yes
47	SG092036	Split/Splitless Tapered FocusLiner	FocusLiner	3.4 mm	5 mm	54 mm	500 µL	Glass	Quartz	Yes
48	SG092037	Split/Splitless Focusliner	FocusLiner	3.4 mm	5 mm	54 mm	500 µL	Glass	Quartz	Yes
49	SG092038	Split/Splitless Liner	Gooseneck	3.4 mm	5 mm	54 mm	500 µL	Glass		Yes
50	SG092039	Splitless Liner with Taper	Tapered	2 mm	5 mm	54 mm	170 µL	Glass		Yes
51	SG092113	Splitless Liner with slots	Straight	2 mm	6.3 mm	72 mm	250 µL	Glass		Yes
52	CP14988	LVI Liner filled with deactivated Chromosorb WAW 60 - 80 mesh	Gooseneck	3.4 mm	5 mm	54 mm	500 µL	Glass	Quartz	Yes

1177 Injector

Parts and Supplies		
Ceramic scoring wafer (for cutting fused silica columns)	0190015800	
Capillary column nut	0394955100	
Ferrule, no hole	2869459001	10/pk
Septum pick	720000CP-8400	
Siltek Deactivated Restek Injector Liners		
Siltek™ 4 mm ID fritted Injector insert for split injections (must be used in combination with PFPD detector)	RT210462145	5/pk
Siltek™ 4 mm ID, unpacked, single gooseneck glass Injector inserts for split/splitless injections	RT207992145	5/pk
Siltek™ 4 mm ID, unpacked, open glass Injector inserts for split/splitless injections	RT207732145	5/pk
Siltek™ 4 mm ID, unpacked, open glass Injector inserts for splitless injections	RT207132145	5/pk
Deactivated Varian Injector Liners		
Injector Liner unpacked, 4 mm ID, similar to HP # 18740-80190	392611931	5/pk
Injector Liner, packed W/10% OV-1 on Chromosorb-W HP 4 MM ID, similar to HP # 18740-60840	392611933	5/pk
Injector Liner, packed with FS wool, 4 MM ID, similar to HP # 18740-80190, MODIFIED	392611932	5/pk
Split Injector Liner, 4 MM ID, packed with FS wool, similar to HP # 19251-60540	392611934	5/pk
Double gooseneck Splitless Injector Liner, 4 MM ID, similar to HP # 5062-3587	392611929	5/pk

1177 Injector

Deactivated Varian Injector Liners

Gooseneck split/splitless Injector Liner, 2 MM ID, modified 2 MM	392611926	5/pk
4 mm ID, wool plug, single gooseneck glass Injector inserts	392611936	5/pk
Gooseneck splitless Injector liner, 4 MM ID, packed with FS wool, similar to HP # 5181-3316	392611928	5/pk
4 mm ID, unpacked, single gooseneck glass Injector inserts, similar to HP # 5181-3316	392611927	5/pk
4 mm ID, wool plug, open glass Injector inserts	392611937	5/pk
4 mm ID, unpacked, open glass Injector inserts, similar to HP # S 18740-80220, 5181-8818	392611925	5/pk
2 mm ID, wool plug, open glass Injector inserts	392611938	5/pk
2 mm ID, unpacked, open glass Injector inserts, similar to HP # S 18740-80220, 5181-8818	392611924	5/pk
SPME insert, 0.75 mm ID for SPME fiber injections	392611998	5/pk

Deactivated SGE Injector Liners (HP model 5890, 6890, 6850, 4890 GCS)

SGE direct inlet Liner, 1.2 MM ID, Straight-Through	SG092016	5/pk
SGE split Injector liner 4 MM ID with quartz wool	SG092001	5/pk

1177 Injector

Deactivated SGE Injector Liners (HP model 5890, 6890, 6850, 4890 GCS)		
No Picture available SGE split inlet Liner, 4 MM ID, Straight-Through	SG092007	5/pk
No Picture available SGE split/less 2.3 MM ID fast focusliner™ inlet liner	SG092005	5/pk
 SGE split/less 4 MM ID tapered focusliner™ inlet liner	SG092003	5/pk
 SGE split/less focusliner™, 4mm ID	SG092002	5/pk
 SGE split/splitless 2.3 MM fast tapered focusliner™ inlet liner	SG092111	5/pk
No Picture available SGE split/splitless inlet liner, 4 MM ID, double taper	SG092018	5/pk
 SGE split/splitless inlet liner, 4 MM ID, recessed gooseneck W/ Quartz wool	SG092010	5/pk
 SGE split/splitless inlet liner, 4 MM ID, Single Taper	SG092017	5/pk
 SGE split/splitless inlet liner, 4 MM ID, Single Taper W/ Quartz wool	SG092019	5/pk
 SGE splitless inlet liner, 2 MM ID, recessed gooseneck	SG092013	5/pk
No Picture available SGE splitless inlet liner, 2 MM ID, Straight-through, Quartz	SG092004	5/pk

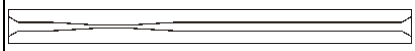
1177 Injector

Septa	9.0 mm BTO Centerguide™ septa	CR298713	50/pk
	9.0 mm Advanced Green 3 Centerguide™ septa	CR246713	50/pk
	9.0 mm Marathon Centerguide™ septa	CR239778	25/pk
Insert Seals	Viton O-ring for 6.3 and 6.5mm OD liners, 260 °C max	8850103100	25/pk
	Graphite O-ring 6.5 mm ID to fit HP Splitless 6.5 mm OD Liners	392611930	10/pk
	Graphite O-ring 6.5 mm ID, fits 6.5 mm OD Liners	392611940	10/pk
	Graphite O-ring 6.3 mm ID, for HP Split 6.3 mm OD Liners, similar to HP # 5180-4168	392611935	10/pk

1078/1079 PTV Injector

Parts and Supplies		
Ceramic scoring wafer (for cutting fused silica columns)	0190015800	
Capillary column nut	0394955100	
Ferrule, no hole	2869459001	10/pk
Septum pick	720000CP-8400	

SPME Mode Insert		
Insert, 0.8 mm SPME for Fiber Injections	392611948	5/pk

On-Column Mode Inserts		
 Insert, tapered for column sealing with 0.53 mm ID capillary columns	190010907	5/pk
Insert, high performance for column sealing with 0.32 or 0.25 mm capillary columns	190010906	5/pk

Split Inserts		
3.4 mm fritted Injector insert for enhanced mixing for linear split performance	392611946	5/pk

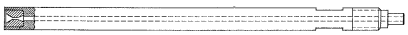
1078/1079 PTV Injector

Split Inserts		
3.4 mm unpacked Injector insert	392611945	5/pk
3.4 mm packed w/10% OV101 on Chromosorb for full range of capillary columns	392611954	5/pk
Splitless Mode Inserts		
2.0 mm open insert to minimize dead volume	392611947	5/pk
2.0 mm packed insert with glass wool for split/splitless injections	392611953	5/pk
0.5 mm open insert for trace analysis and thermolabile compounds	392611949	5/pk
1078/1079 PTV Deactivated SGE Injector Liners		
SGE LVI Large volume Injection inlet liner, sintered glass	SG092245	5/pk
SGE Split/less 3.4 mm ID Tapered Focusliner inlet liner	SG092036	5/pk
SGE Split/Splitless 3.4 mm ID Focusliner inlet liner	SG092037	5/pk
SGE Split/Splitless 3.4 mm ID inlet liner, single taper	SG092038	5/pk
SGE Splitless 2 mm ID inlet liner, single taper	SG092039	5/pk

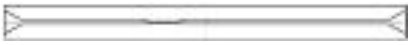
1078/1079 PTV Injector

Split Inserts			
	SGE Straight-Thtough inlet liner, 0.5 mm ID	SG092031	5/pk
Septa	11.5 mm BTO Centerguide™ septa	CR298777	50/pk
	11.5 mm Advanced Green 3 Centerguide™ septa	CR246725	50/pk
	11.5 mm Marathon Centerguide™ septa	CR239787	25/pk
Insert Ferrule	1079 PTV graphite insert ferrule	392534201	10/pk

1041/1061 Injector

Parts and Supplies			
Septa	Septa BTO, 9.5 mm	CR298705	50/pk
	Septa Marathon, 9.5 mm	CR239188	25/pk
	Septa, Advanced Green 3, 9.5 mm	CR246124	50/pk
Inserts			
	1041 Insert for 0.53 mm ID columns	392543101	
	1060/1061 Flash Injector Liner, 0.53 mm ID Capillary columns	392611943	5/pk
	1060/1061 Flash Injector Liner, W/Glass wool, for packed columns	392611944	5/pk
No Picture available			
	1041 Stainless Steel Insert for Headspace injection	CP742552	1/pk
Packed Column Adapter Kits			
Packed Column Adapter Kit for 1041, 1/8” SS columns		03-925588-91	
Packed Column Adapter Kit for 1061, 1/8” SS columns		03-925588-92	
Packed Column Adapter Kit for 1041& 1061, 1/4” glass columns		03-925586-91	
Note that these kits contain Injector and detector adapters			

1093 Injector

Parts and Supplies			
Septa	7/16" (11.5MM) HT SEPTA,TEFLON FACED	00-997630-02	25/pk
	BTO Centerguide™ septa, 11.5 mm	CR298777	50/pk
	Marathon, Centerguide™ septa, 11.5 mm	CR239787	25/pk
	Advance Green 3, Centerguide™ septa, 11.5 mm	CR246725	50/pk
	Heater/Probe Harness, 120 Volt AC	391833400	
	Heater/Probe Harness, 240 Volt AC	391833401	
	Septum Support	391821100	
	Injector Switch Actuator	390820601	
	Union 1/8” W/Flow Restriction	3600004601	
	1/8” Swagelok brass plug	2824707101	10/pk
	Injector Nut W/ Cage	394966601	
Column Ferrule	Polymide/Graphite 0.5mm ID	2869458101	10/pk
	Polymide/Graphite 0.8mm ID	2869458200	10/pk
	Graphite 0.5mm ID	SG072626	10/pk
	Graphite 0.8mm ID	392611909	10/pk
Inserts			
	SPI 0.5mm High Linearity direct injection liner for 0.25mm and 0.32 mm ID columns	190010906	5/pk
	SPI High Linearity On-column injection liner for 0.53mm ID columns	190010907	5/pk
	SPI Injector direct injection liner, 0.8mm ID, packed Glass wool, injection volumes> 1 ql	392611950	5/pk
No Picture Available	0.5mm ID, SPI deactivated Injector liner	SG092027	5/pk
No Picture Available	0.8mm ID, 0.25mm restriction SPI deactivated Injector Liner	SG092030	5/pk
No Picture Available	0.8mm ID, 0.5mm restriction SPI deactivated Injector Liner	SG092034	5/pk

Detectors	
Flame Ionization Detector (FID)	
Flame Tip Assembly (0.02″ ID)	02-001938-00
Aluminum Crunch Washer (25)	15-003347-01
Thermionic Specific Detector	
TSD Bead Probe	03-906074-00
TSD Bead Probe, preconditioned	03-906074-01
Electron Capture Detector (ECD)	
ECD Cell with ^{63}Ni Foil	02-001972-01
ECD Wrench	72-000081-00
Thermal Conductivity Detector (TCD)	
TCD Make-up Gas Option	03-925605-01
Pulsed Flame Photometric Detector (PFPD)	
Aluminum Crunch Washer	15-003347-00
Combustor, Quartz (2mm ID)	03-925176-00
Combustor, Quartz (3 mm ID)	03-925177-00
Filter Assembly, Sulfur	03-925151-01
Filter Assembly, Phosphorus	03-925151-02
Igniter Assembly	03-925160-00
Combustor support, 2 mm	03-925178-00
Combustor support, 3 mm	03-925179-01
Combustor support seal	03-925138-00

TECHNICAL TIPS

This section will give technical tips regarding common questions like:

1. Chromatographic Interpretations, on page 458.
2. To Bleed not to Bleed ….. that is the question [1], on page 461.
3. Choosing the right GC Injection Technique [2], on page 466.
4. GC line Labyrinth – How to choose the right Injector Insert [3], on page 472.

[1] Source Technical helpdesk Europe, Middelburg, The Netherlands
[2] Source Technical helpdesk Europe, Middelburg, The Netherlands
[3] Source Technical helpdesk Europe, Middelburg, The Netherlands

CHROMATOGRAPHIC INTERPRETATIONS

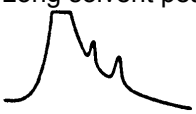
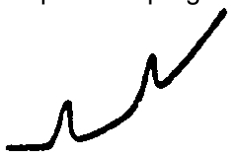
The troubleshooting procedures outlined in this section are structured to accommodate deviations from known responses of a standard sample, including peak shapes, peak areas, retention times, baseline characteristics, etc.

In these cases, the general characteristics of the chromatogram are well known and even slight deviations can be discerned.
A vast majority of gas chromatographic malfunctions will be indicated by one or more of the following symptoms; inspect the chromatogram for all of the symptoms:

- Change in peak size (peaks of different area, or height than expected).
- Change in retention times.
- Change in baseline stability.
- Asymmetrical peak shapes (non-Gaussian, skewed, etc.).
- Change from expected peaks.

For overall troubleshooting of the capillary system based on the appearance of the chromatogram, refer to Table Capillary Chromatographic Interpretations.
If a problem exists which cannot be identified and/or corrected, call your nearest Varian Service Center for assistance.

Symptom	***Cause***	***Remedy***
Multiple peaks from a single compound	Injection is too large. Initial temperature is too low.	Reduce the size of the injection. Initial temperature should be no more than 20°C below the solvent boiling point for samples greater than or equal to 1.0 mL.
Tailing	Degraded column or sample adsorption. Frit may be clogged. Contaminated insert. Excess dead volume. Injector not heating correctly.	Replace or recondition column. Replace purge frit or use needle valve. Replace glass insert. Recheck Injector assembly and column connections. Check make-up gas flow. Check Injector heating/cooling functions.
Peak broadening, poor resolution	Degraded column. Wrong carrier gas flow.	Replace or recondition column.

Symptom	***Cause***	***Remedy***
		Check carrier gas flow.
Ghost peaks during temperature programming, no injection made	Contaminated Injector.	Replace or clean Injector insert. Bake out Injector per Section 4.
Leading peak	Overloaded column. Carrier gas flow restricted.	Decrease sample size or increase dilution; use column with thicker film. Check column connections and Injector insert for obstructions.
Long solvent peak tail 	Injector temperature too high. Injector leakage. Column temperature too high.	Lower initial Injector temperature. Reseat column to glass insert. See Section 3. Decrease column temperature.
No sample peak or peak too small	Leak at column inlet. Syringe is leaking. Broken column. Injector is leaking.	Check for leaks (Section 4, paragraph 4.2). Repair or replace syringe. Replace or repair column. Check or replace septum or Injector.
Rising baseline when temperature programming 	Column temperature limit is exceeded. Contamination.	Reduce maximum temperature Use column with appropriate coating thickness for sample. For relatively non-volatile compounds that require a high retention temperature, choose a 0.1μ coating thickness. Check gas filters. Correct all leaks, checking for the following: (a)make-up gas line union (b)column-to-detector connection (c)detector insert-to-detector crossover connection (d)detector crossover-to-

Symptom	***Cause***	***Remedy***
	Instrument problem.	detector body connection Condition the column or bake out the column as described in Section 4. In badly contaminated systems, it may be necessary to lift the detector crossover from the ion oven, then heat the lines and crossover with a flame while purging with an inert gas. Install an uncoated test column and repeat the temperature program.
High boiling compounds, C_{60} through C_{80}, not eluting	Wrong flame tip.	Change to ceramic flame tip, P/N 03-918351-00.
Syringe hits insert	Glass insert installed upside down (wrong end facing up).	Reverse glass insert.

TO BLEED OR NOT TO BLEED … THAT IS THE QUESTION

As GC and capillary column manufacturer Varian is regularly confronted with customer queries with respect to rising baselines and baseline instability. These effects are often attributed to column failures and unacceptable column bleed is the diagnosis. But is this really the case? Here follows a short guide into more effective troubleshooting on the baseline.
A rising baseline is part of many temperature-programmed analyses in gas chromatography. The major factors contributing to this rise are:

1. Stationary phase bleed from the column.
2. Contamination in the Injector system.
3. Contamination of the detector system.
4. Carrier gas impurities.

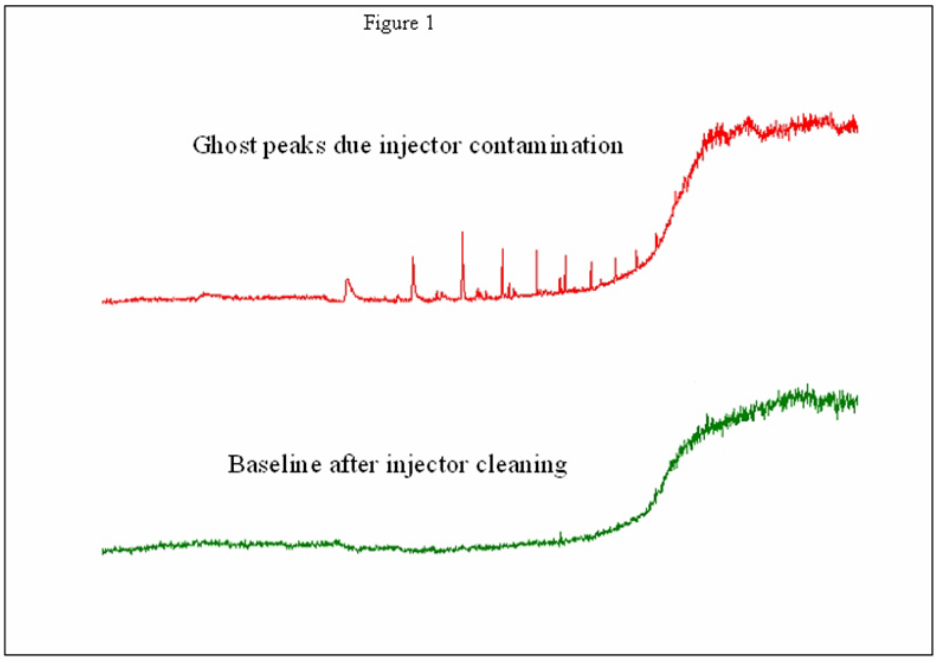
Both the column and the Injector/detector system can play a role in baseline rises and it is important to distinguish between the 2 sources.
Starting the troubleshooting exercise by immediately disconnecting the column from Injector and/or detector can mean an unstable system for at least a couple hours. It can be more effective to have a closer look at the baseline itself first.

The baseline below 250 °C

Improvements in column technology and stationary phase synthesis during the last few years have resulted in highly temperature stable capillary columns. Examples of these are the Varian Factor Four column range. These columns only show some measurable bleed levels close to their maximum operating temperature. This maximum temperature is often around 325 °C. The bleed level of a capillary column roughly doubles with every 14 °C and it means that already at oven temperatures 75 °C below the maximum temperature its bleed level is virtually zero. Therefore, any significant baseline rise starting before 250 °C can safely be attributed to system contamination from medium to high molecular weight residues. Most frequently these are originating from a contaminated Injector. This contamination may use the column as a transport device but the column is not the primary source. Major air leakage however, may oxidize the liquid phase at temperatures lower than 250 °C and high column bleed level can results of this.

Injector contamination

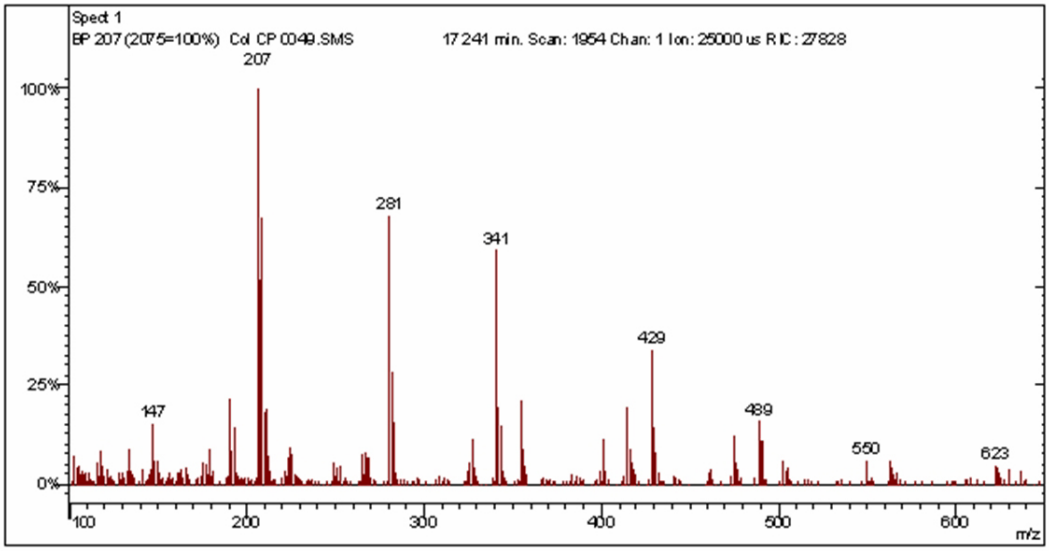
Unstable and noisy baselines, which include ghost peaks during a blank analysis run, are hardly ever caused by column bleed.



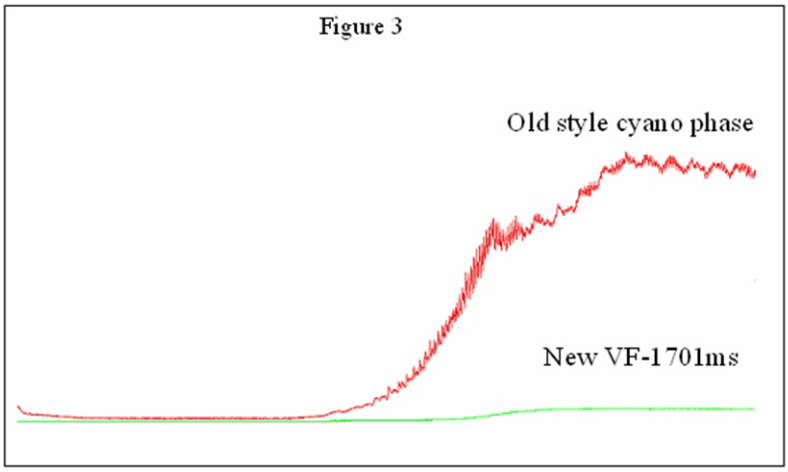
Clear distinctive peaks eluting during such a blank analysis run are an indication again of Injector contamination (figure 1).
Higher molecular weight residues inside the Injector are continuously being introduced on to the column, trapped there at lower oven temperatures and eluted again at higher temperatures. Injector contamination can be confirmed by lowering the Injector temperature to e.g. 100 °C. Evaporation from the Injector of the higher molecular residues is significantly reduced at these lower temperatures. The overall bleed level should drop and the majority of the ghost peaks should disappear. In case of a split/splitless injection Injector contamination can also be confirmed by extending the splitless time period considerably. A contaminated Injector will expose itself by an increased bleed level and/or higher ghost peaks. If the Injector is contributing to the bleed level maintenance should be performed. In particular replace the septum, liner and glass wool. Confirm the cleanliness of the Injector by performing a blank injection.
If lowering the Injector temperature has little effect on the total bleed level the focus of attention should be directed towards the column or to the purity of the carrier gas.

The Column

Mass spectra taken from the bleed can be of great help in determining its source. The main bleed products from all siloxane-based columns are some smaller cyclic siloxane fragments formed by degradation of the liquid phase. These fragments have m/z ions of 207, 281 and 341 (figure 2). However, the presence of these ions in the spectrum is no confirmation of column bleed. Also the septum is made of almost 100 % pure polydimethylsiloxane just like the liquid phase of the capillary column. The total siloxane amount of a septum can be more than a factor 40 of that present in a capillary column. Torn away pieces of septum inside the liner or septum bleed can therefore be responsible for this typical mass spectrum. In cases where the mass spectra of the bleed is dominated by other m/z ions than 207 and 281 the cause of the bleed is most likely Injector contamination due to sample residues. Degradation of the liquid phase and formation of bleed products is often the result of low ppm level presence of oxygen and moisture in the carrier gas. It is therefore imperative to install high quality gas filters close to the GC.



Bleed formation from the column is a continuous process and usually exhibits itself as a slow steady climb of the baseline. Exceptions to this general rule are the older type cyanopropyl containing liquid phases. These columns may form a wavy, sinusoid like, rising baseline close to their maximum operating temperature.



Phase degradation at these temperatures is creating bleed products and they are trapped on the column during the rapid cool-down of the oven at the end of the analysis. These relatively small, low molecular weight degradation products are subsequently eluted during the next analyses. It can sometimes be difficult to distinguish this bleed pattern from a contaminated Injector induced bleed. Lowering the Injector temperature will not reduce this type of bleed
The problem can be partially solved by a more controlled and slower cool down of the oven. Recent implementation of Varian's FactorFour technology in the cyano-type phases like the VF-1701ms and VF-624ms have effectively eliminated these problems al together so that the column bleed patterns and levels are like any other MS grade column. Wave like baselines patterns, which already begin at lower temperatures, can be the result of a poorly conditioned column but can also be related to unstable column flows or impurities in the carrier gas or detector gases.

Gas Impurities

Impurities in either the carrier gas or detector gases can cause unstable baselines.

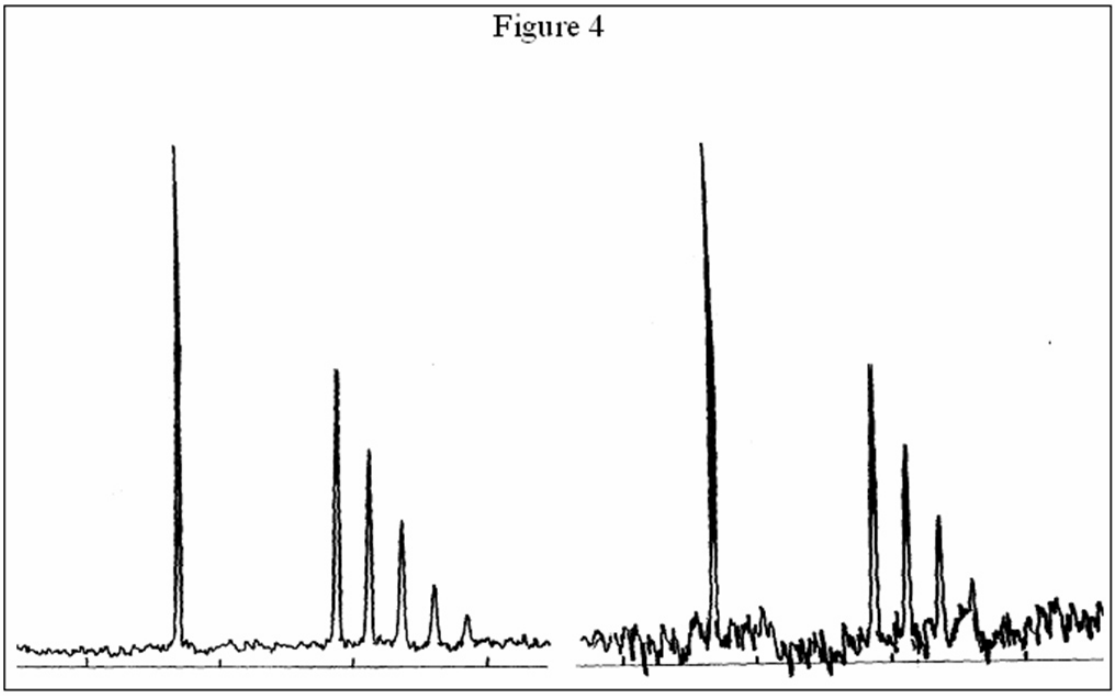


Figure 4 shows the effect on the baseline stability due to the presence of some hydrocarbon impurities in the hydrogen gas flow for an FID. A noisy baseline caused by gas flow impurities is independent of oven temperatures and visible throughout the entire temperature program.
The installation of a good quality gas purifying system like the Varian Gas-Clean filters will maintain a supply of high quality gasses to both the column and detector. Overall baseline stability will improve. If positioned close enough to the GC such a system will guarantee oxygen, moisture and hydrocarbon free gas supply regardless of initial gas bottle quality or the possible presence of leakage anywhere in the gas supply system.

CHOOSING THE RIGHT GC INJECTION TECHNIQUE

A vast number of injection techniques are being used in gas chromatography today. Some have been around since the early days of GC while the popularity of others is sometimes of more recent date. This article attempts to briefly characterize the most commonly used techniques, their applications and their limitations.
The analysis in GC starts with the introduction of the sample onto the column. It is also by far the most critical step in the entire analysis. The liquid sample is vaporized either inside the column itself or in a vaporization chamber positioned just in front of the column.
The injection system should fulfill the following requirements:

- The injected amount should not overload the column.
- The injection bandwidth should be small compared to the band broadening effects in the capillary column.

Other requirements, which could be applied, are:

- The column should be able to achieve its optimum separation efficiency.
- Sample composition should remain intact. No discrimination should occur on basis of boiling point, polarity, concentration or thermal stability.
- Applicable to trace analysis as well as high concentration samples.
- The injected amount should be reproducible.

No injection technique will meet all of the above demands. Some requirements will have to be sacrificed in order to meet the others. The detection of traces and higher concentrations both at the same time is often difficult to combine within one single injection technique. The choice of injection technique is often determined by the concentration levels, which have to be reached.

- Direct Injection
- Split Injection
- Splitless Injection
- Cool on-column
- Programmed Temperature Vaporizer (PTV)

Direct Injection

Description	Sample is vaporized in the hot insert. The entire sample enters the column.
Volume(s)	0.1 µL – 1 µL
Concentration Range	1 ppm - %-levels.
Application	Applicable to packed and 0.53 mm ID capillary columns No refocusing on the column is needed.

Direct injection is the most commonly used injection technique for packed columns and 0.53 mm ID columns. Its major advantages are its ease of use and the wide concentration range that it can cover. It is especially suitable for the lower concentrations in the ppm range, which are below the scope of the split injection. Higher gas flow rates are needed for an efficient and quick sample transfer from the Injector liner to the column. The minimum gas flow for direct injection is about 5 mL/min. Direct injection can therefore only be applied to columns, which can cater for higher column flow rates such as, packed columns or 0.53 mm ID columns. These columns can also handle the higher concentration levels because of their increased sample capacity. Direct injection will produce poor peak shapes if too large injection volumes (> 1 µL) are applied. Discrimination effects are low compared to split injections. Direct injection is sometimes confused with the splitless type injection described further below. The Varian Injector **models 1040/1041** and **1060/1061** are designed for various types of direct injection.

Split Injection

Description	Sample is vaporized in the hot insert. Only a fraction of sample enters the column. The majority is vented of through the split vent.
Volume(s)	0.1µL – 1 µL
Concentration Range	50 ppm - %-level
Remark	Applicable to all capillary columns. Usually no refocusing on the column is needed.

Beyond doubt the most widely used injection technique for capillary columns. This is despite its tendency for discrimination of compounds with higher boiling points. The reproducibility of the injection is strongly dependent on liner geometry and heat capacity. (See also the Tech Tip: **the jungle of injection liners,** *e-inspirations*, No. 3, September 2004). Sample discrimination during the evaporation from the syringe also occurs due to differences in component volatility. The difference in sample capacity of the various capillary columns

(varying internal diameters and film thickness) can easily be addressed by changing the split flow. The relatively high split flow takes care of a quick and efficient sample introduction onto the column. Split injections can exploit the full separation power of the capillary column because of this quick sample introduction. The following table gives some general guidelines, which split-flows should be applied to which column diameter. Split flows and split ratios are related to column sample capacity and the minimum flow needed in the Injector to minimize band-broadening effects.

Column ID [µm]	*Split ratio*	*Minimum split flow [mL/min]*
530	1:5 to 1:15	15
320	1:20 to 1:250	25
250	1:50 to 1:250	40
150	1:150 to 1:500	75
100	> 1:500	150

The suggested minimum split ratios and split flows can result in band broadening inside the Injector liner and therefore loss of separation efficiency is likely.
The **1177 Injector** model is the most recent split/splitless Injector of Varian.

Splitless Injection

Description	Sample evaporates in the hot Injector. The majority of the sample enters the column during the first 1 – 2 min of the analysis.
Volume(s)	0.5 µL – 2 µL
Concentration Range	0.5 ppm – 50 ppm
Remark	Refocusing on the column needs to be supported by retention gaps and low oven temperatures.

The most widely used technique for low ppm level samples. Splitless is often confused with direct injection. Sample is being introduced on the column during the entire splitless time. Due to this 1 – 2 min period there is a large initial injection bandwidth and refocusing of the analytes on the column is essential in order to obtain good symmetrical narrow peak shapes. This is done in 2 ways:

1. Cold trapping. The large temperature drop between Injector (250 °C) and a low initial oven temperature (50 °C) effectively reduces the mobility of higher boiling compounds to virtually zero. These compounds freeze in a narrow band and only start to migrate again during the temperature program. This cold trapping effect combines both focusing due to thermal condensation as well as focusing as a result of a strong retention in the columns stationary phase.

2. Solvent focusing. Reconcentration of lower boiling components (close to the boiling point of the solvent) takes place by the solvent effect. Low oven temperatures will allow the solvent to condense in the column together with the low boiling sample components. The liquid film formed by the solvent will start to evaporate and the sample components will concentrate in a continuously smaller liquid film, resulting in a narrow band of concentrated sample components. Whether this process really takes place as described here is still a point of discussion among chromatographers. Fact is that both choices of solvent and initial oven temperatures are important in obtaining narrow peak shapes. The table below provides some general guidelines.

Solvent	Boiling Point [°C]	Suggested Initial Oven Temperature [°C]
n-Pentane	36	10 to ambient
Dichloromethane	36	10 to ambient
Carbon disulfide	40	10 to ambient
Chloroform	46	10 to ambient
Methanol	61	25
n-Hexane	65	35
Ethyl acetate	69	40
Acetonitrile	77	45
n-Heptane	82	50
Isooctane	98	70

Column flow and the volume of the liner determine the speed and effectiveness of the sample transfer from Injector to column. Very low column flows prevent the use of splitless and it is mainly for this reason that 100 µm are not suited of splitless type of injections. 150 µm columns can be used but only if the splitless time period is long enough and the liner volume limited to 250 µL to permit adequate sample transfer.
Splitless is not very suitable for samples where the components of interest elute closely to the solvent either in front or behind. Insufficient focusing of the analytes will produce poor peak shapes. In those cases a direct injection on a 0.53 mm ID column is often preferable.
The **1177 Injector** model is the most recent split/splitless Injector of Varian.

Cool on-column

Description	Sample passes from the syringe into the column. The column is kept cool during injection and is subsequently heated. The Injector is kept cool during sample introduction.
Volume(s)	0.1 µL – 2 µL
Concentration Range	0.25 ppm – 50 ppm
Remark	Refocusing on the column needs to be supported by retention gaps and low oven temperatures.

Cool on-column is perhaps the most “ideal” injection technique. It eliminates inlet-related discrimination and alteration and combines this with a high analytical precision. Syringe discrimination, as experienced with split and splitless, is absent. Components eluting just in front of the solvent however are difficult to focus and therefore difficult to determine. Cool on column follows closely the splitless injection type with respect to refocusing aspects like oven temperatures and the use of retention gaps. The market acceptance of the cool on-column Injector has always been rather limited. This is mainly due to the instrumental difficulties during the early days of its introduction. Its competitor splitless is far easier to automate and this is one of the reasons for its wide acceptance. Automated on-column Injectors require a 0.53 mm ID piece of deactivated fused silica or retention gap as inlet piece because of the gauge 26 syringes used in auto samplers. Although not a cold on-column Injector in the true sense, the Varian **1093 SPI Injector** covers the same application range and provides similar results.

Programmed Temperature Vaporizer (PTV)

Description	Sample passes as a liquid from the syringe into a cooled inlet. The sample is subsequently heated to vaporize the sample. Split or splitless injection can be applied.
Volume(s)	0.1 µL – 250 µL
Concentration Range	1 ppb – 50 ppm
Remark	Refocusing on the column needs to be supported by retention gaps and low oven temperatures.

The PTV Injector can arguably be considered as the most universal of Injector, capable of handling a wide variety of sample types, concentrations and volumes. In essence, it is designed as a split/splitless Injector that can be rapidly heated (and cooled. The syringe introduces the sample in it liquid state into a cold Injector insert. Discrimination of the sample compounds by additional evaporation of the more volatile constituents is absent. Evaporation of

components starts as the insert is being heated. The PTV can operate in various modes:

Cold Split Injection

… is comparable with a split injection but with a cold transfer from syringe to insert. This provides less discrimination effects and therefore more accurate results. Refocusing using retention gaps and low oven temperatures may be necessary for lower boiling compounds.

Cold Splitless Injection

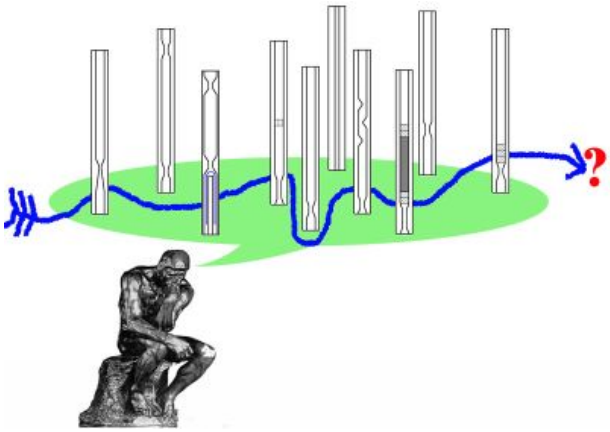
… is similar to hot splitless. It has the same advantages as cold split with respect to the cold transfer from the syringe. The classic hot splitless technique can result in thermal degradation due to the long residence time of components in the hot insert. The PTV in cold splitless mode also avoids this phenomenon. Refocusing using retention gaps and low oven temperatures may be necessary for lower boiling compounds.

Solvent Venting

… permits the injection of large sample volumes (250 µL). It enables detection limits in the ppb range. The majority of the solvent is removed through the split vent. The components are retained in the liner by cold trapping, aided in some cases by packed supports. At the end of the vent period the split vent is closed and the insert is rapidly heated. This technique requires a slow sample introduction in order to avoid overloading the insert. Lower boiling compounds will unavoidably be partly vented of together with the solvent depending on insert temperatures, type of packed support and vent times. In general, light boiling solvents are preferred for better results. Some column refocusing on the column supported by retention gaps and low initial oven temperatures is needed.

The Varian **1079 PTV Injector** can perform all of these modes.

GC LINER LABYRINTH - HOW TO CHOOSE THE RIGHT INJECTOR INSERT



Injections and vaporization in gas chromatography are influenced by a number of variables. The liner always plays a significant role. The most important ones are liner design and volume, sample composition, Injector heat capacity, heat transfer and adsorption effects. These factors together with the type of injection determine for a large extent the choice of liner.

Liner Types

Three major types of injection based on flash vaporization of the sample can be distinguished:

1. ***Direct injection***
   After flash vaporization the entire sample is transferred to the column. The sample transfer speed in the Injector is determined by column flow and volume of the Injector liner. This injection type is most commonly applied for 0.53 mm ID columns or packed columns. Injection volumes usually do not exceed 1 µL. This maximum in injection volume is related to the internal volume of the liner.

2. ***Split injection***
   After flash vaporization the sample is divided into 2 streams. One directed towards the column, the other to the split vent. Here, the sample transfer speed in the Injector is mainly determined by the flow of the split vent and the liner volume. The column flow plays an insignificant role. A high transfer speed of the sample is critical for the quick introduction of the compounds into the capillary column maintaining the columns separation efficiency Split injections are used for all capillary columns with ID‘s ranging from 100 µm to 530 µm. The maximum injection volume for split injections is about 1 µL.

3. ***Split/Splitless***
   During the initial splitless period the vaporized sample is transferred to the column. The capillary column flow and the liner volume determine the speed and efficiency of this process. Larger injection volumes are possible for splitless type injections; volumes up to 5 µL are not uncommon. These large injection volumes can never be injected at once because of possible liner overload but must be careful introduced at a rate of about 1 µL/sec.

Liner volume

The sample gas volume, which at one time resides in the Injector, should in all cases be smaller than the internal volume of the liner. If not, the liner is overloaded and back flash of the sample into various (metal) parts of the Injector may occur. This phenomenon will cause poor injection profiles, bad peak shapes, adsorption and possible sample carry over. Many liners used for direct and split injection have dimensions of about 80 mm x 4 mm resulting in an internal volume of ca. 1mL. The effective volume available for the sample is however significantly lower as part of the liner volume will always be filled with carrier gas. The list below indicates the solvent expansion volume per µL at 250 °C and 50 kPa

Methanol	0.65 mL
n-hexane	0.2 mL
Isopropanol	0.34 mL
Water	1.46 mL
Dichloromethane	0.41 mL

It is clear from the above that water and methanol in particular quickly can cause liner overload and some care must taken if these solvents are used. For these solvents smaller injection volumes than 1 µL are to be recommended.

Injector heat capacity

The efficient and quick evaporation of the sample is influenced by the following factors:

- sample volume
- type of solvent and sample components
- Injector temperature
- Injector and liner geometry and design
- presence of quartz wool or other liner fillings

The last three determine the heat capacity of the Injector and its ability for heat transfer to the sample. The below list compares the amount of energy needed for evaporation of methanol and water relative to cyclohexane.

Water	5.22
Methanol	3.29
Cyclohexane	1.00

Solvents like methanol and water require much more energy than other common solvents for their evaporation therefore they need a larger heat capacity and better heat transfer system for a quick evaporation.

This can be accomplished by using higher Injector temperatures (>275 °C) and larger liner surface areas. Glass or quartz wool is often used to increase the Injectors heat transfer capacity. The evaporation of higher boiling compounds also requires more energy and also in those cases quartz wool is often used. Besides benefits glass or quartz wool also has a few disadvantages. The wool can become adsorptive especially if some fibers are broken or when it has become dirty. It should be exchanged at a regular basis to prevent chromatographic problems

Discrimination effects

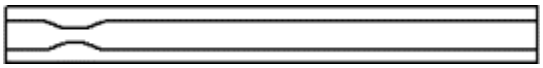
The main aim and challenge of injections is to maintain sample integrity and sample composition during its transition from the liquid to the gaseous state and also during the sample transfer from Injector to the column. The evaporation process of low boiling compounds is always quicker than that of high molecular weight ones. This difference accounts for the often-mentioned discrimination effects caused by injections. In general, the quicker and more complete the evaporation of the sample, the less discrimination effects will be observed. Higher Injector temperatures, special liner design and quartz wool will help to minimize these effects. Inconsistent discrimination profiles will result in poorer repeatability.

Liners for splitless injections

Straight liner

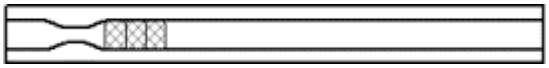
These are low cost liners for samples with a narrow boiling point range and little risk of thermal decomposition. Can be packed with quartz wool to aid evaporation, limit discrimination and trap non-volatile materials. Do not use for high boiling samples. Comes usually in 2 or 4 mm ID. Use the 4 mm version for sample volumes > 2 µL to prevent liner overload.

Gooseneck



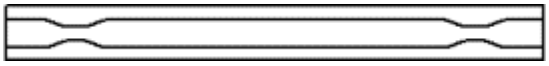
Minimizes breakdown of active compounds and improves the splitless efficiency. The gooseneck prevents the sample from reaching metal Injector parts, which could cause decomposition of sensitive compounds. The gooseneck is positioned at the top of the Injector. It is generally regarded as the best splitless liner.

Gooseneck with wool



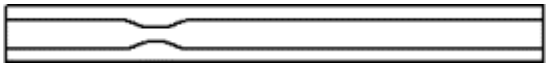
The single gooseneck can be easily packed with quartz or glass wool to minimize discrimination. Wool is also a risk as it can become active and adsorptive due to the trapping of non-volatile residues. Replace it at regular intervals.

Double Gooseneck



The 2 goosenecks isolate the sample vaporization cloud from any metal Injector parts. The risk of catalytic breakdown is reduced. This type of liner can not packed with glass wool.

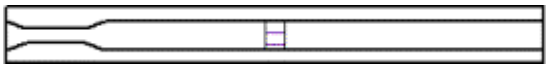
Recessed Gooseneck



This model has the same advantages as the other goosenecks but in addition it can be packed with glass wool at the top. It can also be used for dual column analysis.

Liners for split injections

Frit Splitter



The ceramic frit will prevent dirt and non-volatiles from reaching the column. The frit can become active. The gooseneck prevents back flash.

Inverted -or Laminar Cup Splitter

The cup will promote evaporation and mixing of the sample with the carrier gas. It is the best splitter for higher molecular weight compounds. Slightly larger injection volumes are possible because the cup will hold the sample liquid until it vaporizes. It is difficult to clean and is relatively expensive.

Baffle Splitter

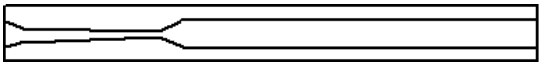
The baffle creates a turbulent flow, which results in more reproducible injections. Not well suited for higher boiling compounds because of incomplete vaporization and thus subject to discrimination.

Liners for direct injections

Straight tube

This type of liner is more suited for packed columns than it is for 0.53 mm ID columns. The wide-bore column should be positioned at the bottom of the liner just piercing through the ferrule. This will minimize the effects of possible dead volume during injections although Injector parts, which are swept poorly with carrier gas will result in poor injection profiles.

Gooseneck



This is a different type of gooseneck liner where the gooseneck holds the wide-bore column in position. This design effectively eliminates any dead volume in the Injector and provides the best possible injection profile for wide-bore columns. In reversed position it can also be used for on-column injections on 0.53 mm columns.

APPENDIX A: ERROR CODES

Code	Error Description	Error level	Cause Condition(s)	450-GC action Error Led on for all errors	Action needed
8	Coolant timed out	Event	Column oven standby • EFC21 (gas saver)	Notification Message	Activate method, clear timeout
FID Flame out					
15	Front FID Flameout	Alert	• FID signal is < than given threshold • No gas supply, flow 0.0 mL/min	Notification Message Auto-re-ignite	Ignite FID flame, check EFC flow (gas supply), set FID gasflows
16	Middle FID Flameout	Alert	• FID signal is < than given threshold • No gas supply, flow 0.0 mL/min	Notification Message Auto-re-ignite	Ignite FID flame, check EFC flow (gas supply), set FID gasflows
17	Back FID Flameout	Alert	• FID signal is < than given threshold • No gas supply, flow 0.0 mL/min	Notification Message Auto-re-ignite	Ignite FID flame, check EFC flow (gas supply), set FID gasflows
TCD Air Leak					
18	Front TCD Air Leak	Event	Air Leak	TCD Power off, Electronics off	Check for TCD air-leak
19	Middle TCD Air Leak	Event	Air Leak	TCD Power off, Electronics off	Check for TCD air-leak
20	Back TCD Air Leak	Event	Air Leak	TCD Power off, Electronics off	Check for TCD air-leak
Injector EFC Flow Out of Range					
35	Front EFC Flow Out of Range	Alert/Error[w]	Flow is above max+10% or below -5kPa for more then 10 seconds	Activated Safety Method	Check EFC flows (gas supply)
36	Middle EFC Flow Out of Range	Alert/Error[a]	Flow is above max+10% or below -5kPa for more then 10 seconds	Activated Safety Method	Check EFC flows (gas supply)
37	Back EFC Flow Out of Range	Alert/Error[a]	Flow is above max+10% or below -5kPa for more then 10 seconds	Activated Safety Method	Check EFC flows (gas supply)
Injector EFC Pressure Out of Range					
38	Front EFC Pressure Out of Range	Alert/Error[a]	Pressure is above max+10% or below -5kPa for more then 10 seconds	Notification Message	-

[w] If the used gas is Hydrogen (H_2) the Error level is Error, in all other cases an Alert is given.

39	Middle EFC Pressure Out of Range	Alert/Error[a]	Pressure is above max+10% or below -5kPa for more then 10 seconds	Notification Message	-
40	Back EFC Pressure Out of Range	Alert/Error[x]	Pressure is above max+10% or below -5kPa for more then 10 seconds	Notification Message	-
Injector EFC Flow ready time out					
41	Front EFC Flow Ready Time Out	Alert/Error[a]	Unit not ready for 10 minutes	Activated Safety Method	Check gas supply, set desired pressure, check EFC device, reboot GC
42	Middle EFC Flow Ready Time Out	Alert/Error[a]	Unit not ready for 10 minutes	Activated Safety Method	Check gas supply, set desired pressure, check EFC device, reboot GC
43	Back EFC Flow Ready Time Out	Alert/Error[a]	Unit not ready for 10 minutes	Activated Safety Method	Check gas supply, set desired pressure, check EFC device, reboot GC
Injector EFC Pressure ready time out					
44	Front EFC Pressure Ready Time Out	Alert/Error[a]	Unit not ready for 10 minutes	Activated Safety Method	-
45	Middle EFC Pressure Ready Time Out	Alert/Error[a]	Unit not ready for 10 minutes	Activated Safety Method	-
46	Back EFC Pressure Ready Time Out	Alert/Error[a]	Unit not ready for 10 minutes	Activated Safety Method	-
PFPD Board Failure					
50	Front PFPD Board Failure	Alert	Defect PFDP board	Electronics off (all) H2 flow set to 0.0 mL/min	Replace PFPD board
51	Middle PFPD Board Failure	Alert	Defect PFDP board	Electronics off (all) H2 flow set to 0.0 mL/min	Replace PFPD board
52	Back PFPD Board Failure	Alert	Defect PFDP board	Electronics off (all) H2 flow set to 0.0 mL/min	Replace PFPD board
53	Becoming Not Ready (during run)	Event	-	-	-
54	Becoming Ready (during run)	Event	-	-	-
IOC Failure					
55	IOC Bad Addre Chan	Event	-	-	-

[x] If the used gas is Hydrogen (H_2) the Error level is Error, in all other cases an Alert is given.

56	IOC Bad Cmd	Event	-	-	-
57	IOC Stepper in Motion	Event	-	-	-
58	IOC Bad Parameter	Event	-	-	-
59	IOC Run mode error	Event	-	-	-
60	IOC no Detector	Event	-	-	-
61	IOC Command not Allowed	Event	-	-	-
62	IOC_EFC_BUFFER_OVERFLOW, Unit rebooted	Event	Caused by too many request requested by IOC to EFC Interface print.	GC reboot the interface print firmware	-
63	IOC_EFC_COMMUNICATION_ERROR, Unit Rebooted	Event	Communication between EFC Interface Print and EFC broken.	GC reboot the interface print firmware	-
Zone Open Sensor (zone)					
67	Column Oven Open Sensor	Error	Column Oven sensor not in use, mis used	Electronics off (all) H2 flow set to 0.0 mL/min	Check Column Oven zone
68	Zone 1 Open Sensor	Error	Zone 1 sensor not in use, mis used	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (1)
69	Zone 2 Open Sensor	Error	Zone 2 sensor not in use, mis used	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (2)
70	Zone 3 Open Sensor	Error	Zone 3 sensor not in use, mis used	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (3)
71	Zone 4 Open Sensor	Error	Zone 4 sensor not in use, mis used	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (4)
72	Zone 5 Open Sensor	Error	Zone 5 sensor not in use, mis used	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (5)
73	Zone 6 Open Sensor	Error	Zone 6 sensor not in use, mis used	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (6)
Shorted Sensor (zone)					
74	Column Oven Shorted Sensor	Error	Shorted sensor. Defect Sensor	Electronics off (all) H2 flow set to 0.0 mL/min	Check Column Oven zone
75	Zone 1 Shorted Sensor	Error	Shorted sensor. Defect Sensor	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (1)

76	Zone 2 Shorted Sensor	Error	Shorted sensor. Defect Sensor	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (2)
77	Zone 3 Shorted Sensor	Error	Shorted sensor. Defect Sensor	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (3)
78	Zone 4 Shorted Sensor	Error	Shorted sensor. Defect Sensor	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (4)
79	Zone 5 Shorted Sensor	Error	Shorted sensor. Defect Sensor	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (5)
80	Zone 6 Shorted Sensor	Error	Shorted sensor. Defect Sensor	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (6)
Thermal Runaway (zone)					
81	Column Oven Thermal Runaway	Error	-	Electronics off (all) H2 flow set to 0.0 mL/min	Check Column Oven zone
82	Zone 1 Thermal Runaway	Error	-	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (1)
83	Zone 2 Thermal Runaway	Error	-	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (2)
84	Zone 3 Thermal Runaway	Error	-	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (3)
85	Zone 4 Thermal Runaway	Error	-	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (4)
86	Zone 5 Thermal Runaway	Error	-	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (5)
87	Zone 6 Thermal Runaway	Error	-	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (6)
Max Temper Exceeded (zone)					
88	Column Oven Max Temper Exceeded	Error	Defect Sensor	Electronics off (all) H2 flow set to 0.0 mL/min	Check Column Oven zone
89	Zone 1 Max Temper Exceeded	Error	Defect Sensor	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (1)
90	Zone 2 Max Temper Exceeded	Error	Defect Sensor	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (2)
91	Zone 3 Max Temper Exceeded	Error	Defect Sensor	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (3)

92	Zone 4 Max Temper Exceeded	Error	Defect Sensor	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (4)
93	Zone 5 Max Temper Exceeded	Error	Defect Sensor	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (5)
94	Zone 6 Max Temper Exceeded	Error	Defect Sensor	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (6)
Min Temper Exceeded (zone)					
95	Column Oven Min Temper Exceeded	Error	Turn device off. Temperature below -10 °C, only for non callable devices	Electronics off (all) H2 flow set to 0.0 mL/min	Check Column Oven zone
96	Zone 1 Min Temper Exceeded	Error	Turn device off. Temperature below -10 °C, only for non callable devices	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (1)
97	Zone 2 Min Temper Exceeded	Error	Turn device off. Temperature below -10 °C, only for non callable devices	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (2)
98	Zone 3 Min Temper Exceeded	Error	Turn device off. Temperature below -10 °C, only for non callable devices	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (3)
99	Zone 4 Min Temper Exceeded	Error	Turn device off. Temperature below -10 °C, only for non callable devices	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (4)
100	Zone 5 Min Temper Exceeded	Error	Turn device off. Temperature below -10 °C, only for non callable devices	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (5)
101	Zone 6 Min Temper Exceeded	Error	Turn device off. Temperature below -10 °C, only for non callable devices	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (6)
Too Slow Response (zone)					
109	Column Oven too slow response	Error	Sensor not used (wrong used), mismatch heated device. 230 V GC is using 110 V heaters	Electronics off (all) H2 flow set to 0.0 mL/min	Check Column Oven zone
110	Zone 1 too slow response	Error	Sensor not used (wrong used), mismatch heated device. 230 V GC is using 110 V heaters	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (1)
111	Zone 2 too slow response	Error	Sensor not used (wrong used), mismatch heated device. 230 V GC is using 110 V heaters	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (2)
112	Zone 3 too slow response	Error	Sensor not used (wrong used), mismatch heated device. 230 V GC is using 110 V heaters	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (3)
113	Zone 4 too slow response	Error	Sensor not used (wrong used), mismatch heated device. 230 V GC is using 110 V heaters	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (4)
114	Zone 5 too slow response	Error	Sensor not used (wrong used), mismatch heated device. 230 V GC is using 110 V heaters	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (5)

115	Zone 6 too slow response	Error	Sensor not used (wrong used), mismatch heated device. 230 V GC is using 110 V heaters	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (6)
Too Fast Response (zone)					
116	Column Oven too fast response	Error	Sensor not used (wrong used), mismatch heated device. 110 V GC is using 230 V heaters	Electronics off (all) H2 flow set to 0.0 mL/min	Check Column Oven zone
117	Zone 1 too fast response	Error	Sensor not used (wrong used), mismatch heated device. 110 V GC is using 230 V heaters	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (1)
118	Zone 2 too fast response	Error	Sensor not used (wrong used), mismatch heated device. 110 V GC is using 230 V heaters	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (2)
119	Zone 3 too fast response	Error	Sensor not used (wrong used), mismatch heated device. 110 V GC is using 230 V heaters	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (3)
120	Zone 4 too fast response	Error	Sensor not used (wrong used), mismatch heated device. 110 V GC is using 230 V heaters	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (4)
121	Zone 5 too fast response	Error	Sensor not used (wrong used), mismatch heated device. 110 V GC is using 230 V heaters	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (5)
122	Zone 6 too fast response	Error	Sensor not used (wrong used), mismatch heated device. 110 V GC is using 230 V heaters	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (6)
Fan or Heater					
123	No Fan or Heater detected	Error	Hardware error	-	-
124	Watch Dog Heater activated	Error	Hardware error	-	-
TCD no Air Leak (position)					
230	Front TCD no Air leak	Event	When error (18) is solved within 4 minutes after error (18) appears	Notification Message	-
231	Middle TCD no Air leak	Event	When error (18) is solved within 4 minutes after error (18) appears	Notification Message	-
232	Rear TCD no Air leak	Event	When error (18) is solved within 4 minutes after error (18) appears	Notification Message	-
TCD Air Leak fault (position)					
233	Front TCD Air leak fault	Alert	4 minutes after error (18) where there has been no “No Air Leak”	Turn Electronics and power off	Find source and repair/replace hardware
234	Middle TCD Air leak fault	Alert	4 minutes after error (18) where there has been no “No Air Leak”	Turn Electronics and power off	Find source and repair/replace hardware

235	Rear TCD Air leak fault	Alert	4 minutes after error (18) where there has been no “No Air Leak”	Turn Electronics and power off	Find source and repair/replace hardware
Max Setup Temper Exceeded (position)					
247	Column Oven Max Setup Temper Exceeded	Error	Defect Sensor. Temperature is 10 °C above set temperature limit	Electronics off (all) H2 flow set to 0.0 mL/min	Check Column Oven zone
248	Zone 1 Max Setup temper Exceeded	Error	Defect Sensor. Temperature is 10 °C above set temperature limit	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (1)
249	Zone 2 Max Setup temper Exceeded	Error	Defect Sensor. Temperature is 10 °C above set temperature limit	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (2)
250	Zone 3 Max Setup temper Exceeded	Error	Defect Sensor. Temperature is 10 °C above set temperature limit	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (3)
251	Zone 4 Max Setup temper Exceeded	Error	Defect Sensor. Temperature is 10 °C above set temperature limit	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (4)
252	Zone 5 Max Setup temper Exceeded	Error	Defect Sensor. Temperature is 10 °C above set temperature limit	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (5)
253	Zone 6 Max Setup temper Exceeded	Error	Defect Sensor. Temperature is 10 °C above set temperature limit	Electronics off (all) H2 flow set to 0.0 mL/min	Check zone (6)
Detector EFC Flow out of range					
304	Front DFC Chan1 out of range	Alert/Error[y]	Flow is above max +10 % or below -5 mL/min for more then 10 sec.	Activate Safety Method	Turn off/on flow, check source (gas-supply)
305	Front DFC Chan2 out of range	Alert/Error[a]	Flow is above max +10 % or below -5 mL/min for more then 10 sec.	Activate Safety Method	Turn off/on flow, check source (gas-supply)
306	Front DFC Chan3 out of range	Alert/Error[a]	Flow is above max +10 % or below -5 mL/min for more then 10 sec.	Activate Safety Method	Turn off/on flow, check source (gas-supply)
307	Middle DFC Chan1 out of range	Alert/Error[a]	Flow is above max +10 % or below -5 mL/min for more then 10 sec.	Activate Safety Method	Turn off/on flow, check source (gas-supply)
308	Middle DFC Chan2 out of range	Alert/Error[a]	Flow is above max +10 % or below -5 mL/min for more then 10 sec.	Activate Safety Method	Turn off/on flow, check source (gas-supply)
309	Middle DFC Chan3 out of range	Alert/Error[a]	Flow is above max +10 % or below -5 mL/min for more then 10 sec.	Activate Safety Method	Turn off/on flow, check source (gas-supply)
310	Rear DFC Chan1 out of range	Alert/Error[a]	Flow is above max +10 % or below -5 mL/min for more then 10 sec.	Activate Safety Method	Turn off/on flow, check source (gas-supply)
311	Rear DFC Chan2 out of range	Alert/Error[a]	Flow is above max +10 % or below -5 mL/min for more then 10 sec.	Activate Safety Method	Turn off/on flow, check source (gas-supply)
312	Rear DFC Chan3 out of range	Alert/Error[a]	Flow is above max +10 % or below -5 mL/min for more then 10 sec.	Activate Safety Method	Turn off/on flow, check source (gas-supply)

[y] If the used gas is Hydrogen (H_2) the Error level is Error, in all other cases an Alert is given.

313	Front EFC Channel 1, flow ready time out	Alert/Error[a]	Unit not ready for 10 minutes	-	-
314	Front EFC Channel 2, flow ready time out	Alert/Error[z]	Unit not ready for 10 minutes	-	-
315	Front EFC Channel 3, flow ready time out	Alert/Error[a]	Unit not ready for 10 minutes	-	-
316	Middle EFC Channel 1, flow ready time out	Alert/Error[a]	Unit not ready for 10 minutes	-	-
317	Middle EFC Channel 2, flow ready time out	Alert/Error[a]	Unit not ready for 10 minutes	-	-
318	Middle EFC Channel 3, flow ready time out	Alert/Error[a]	Unit not ready for 10 minutes	-	-
319	Rear EFC Channel 1, flow ready time out	Alert/Error[a]	Unit not ready for 10 minutes	-	-
320	Rear EFC Channel 2, flow ready time out	Alert/Error[a]	Unit not ready for 10 minutes	-	-
321	Rear EFC Channel 3, flow ready time out	Alert/Error[a]	Unit not ready for 10 minutes	-	-
Auto Sampler (AS)					
370	Sampler old firmware	Event	Syringe stepper motor firmware old	-	-
371	Sampler Faulted	Alert	Mismatch AutoSampler	Notification Message	Cable in SID2, check power switch, jumpers on AS, Reboot GC
372	Sampler Communication error	Alert	No communication AS	Notification Message	Check cables, reboot GC
373	Sampler Tower error	Alert	No response AS-tower. Start AS when AS is turned off	-	-
374	Sampler Carrousel error	Alert	Block carrousel when it's turning	-	-
375	Sampler Syringe Sled error	Alert	Block syringe when it's turning	-	-
376	Sampler Plunger error	Alert	Hall sensor unplugged Switched to SPME mode during syringe use	-	Recalibrate AS, reboot GC
377	Sampler Syringe Missing	Alert	No Syringe available	-	Place Syringe
378	Sampler Vail Missing	Alert	No Vial available Vial detect should be turned on	Notification Message	Place Vial in position

[z] If the used gas is Hydrogen (H_2) the Error level is Error, in all other cases an Alert is given.

			(setup)		
379	Sampler Dual Dup Mode Delay Expired	Alert	Delay between runs has expired before the second injection. This error will only appear in dual-/ or duplicate mode	Notification Message?	Remove time out, restart seq.
380	Sampler Run not started after Inj	Alert	Run did not start injection. Contact switch on Injector broken?	Notification Message?	restart
381	Sampler Syringe volume exceeded	Alert	Total Injection volume exceeds syringe volume	Notification Message?	Check error source
383	Sampler Tower sensor not found	Alert	Unplugged sensor: sense tower	-	Check source, solve error
384	Sampler Carrousel sensor not found	Alert	Carrousel not in place	-	Check source, solve error
385	Sampler Syringe sled sensor not found	Alert	Unplugged sensor: sled sensor	-	Check source, solve error
386	Sampler Plunger sensor not found	Alert	-	-	Check source, solve error
387	Sampler Plunger Strokes exceed limit	Alert	Plunger strokes exceed limit	-	Reset plunger stroke counter
Gas saver timeout expired (position)					
388	Front Gas saver timeout exceeded	Event	Gas saver timeout exceeded	Activate standby method	Activated method, remove time out
389	Middle saver timeout exceeded	Event	Gas saver timeout exceeded	Activate standby method	Activated method, remove time out
390	Rear saver timeout exceeded	Event	Gas saver timeout exceeded	Activate standby method	Activated method, remove time out
391	Run ended before second injection	Alert	Stopped	Notification Message?	Check cause, solve, restart automation?
Miscellaneous					
412	Unavailable operation nonautom	Alert	Running, remote with Galaxie	Notification Message?	Check cause, solve, restart automation?
413	IOP_COMMUNICATION_FAILURE_AT_STARTUP_e	Error	IOP Communication Failure at startup	Notification Message?	Replace/fix IOP
425	IOP_SKIPS_ADC_READING_e	Error	IOP too busy (might be due to EFC) and therefore skips ADC reading	Notification Message?	-
Auto Sampler (AS)					
414	Sampler Max Plunger speed exceeded	Alert	Set Injection to 60 µL/se (with testclient) using a 10 µL syringe	Electronics off (all) H2 flow set to 0.0 mL/min	Down load method, reset plunger speed

416	Sampler Tray is missing	Alert	Only on 8410 sampler	Notification Message?	Place tray in position
418	Sampler Vial is unreachable	Alert	Only on 8410 sampler, tray has been turned vial becomes unreachable for the tower	Notification Message	Calibrate AS, place vial, solve error
423	Sampler Vial sensor bad	Alert	Defective sensor	Electronics off (all) H2 flow set to 0.0 mL/min	Replace vial sensor
424	Sampler Column standby timeout expired	Event	Standby timeout expired	Activate method, reset time out	Activate method, reset time out
426	Sampler Plunger stuck on sensor	Alert	(errors not reproducible)	Electronics off (all) H2 flow set to 0.0 mL/min	-
427	Sampler Syringe stuck on sensor	Alert	(errors not reproducible)	Electronics off (all) H2 flow set to 0.0 mL/min	-
428	Sampler Tower stuck on sensor	Alert	(errors not reproducible)	Electronics off (all) H2 flow set to 0.0 mL/min	-
429	Sampler Carrousel stuck on sensor	Alert	(errors not reproducible)	Electronics off (all) H2 flow set to 0.0 mL/min	-
434	Sampler EDS Checksum is bad	Alert	-	-	-
436	IOP Communication failure	Error	IOP Communication failure	Notification Message?	Replace/fix IOP
437	Battery voltage out of range	Event	Battery voltage < 2V or > 4V ➔ error	-	-
439	PGA Calib Failed	Error	IOPSH (measuring system)	-	Recalibrate PGA
440	Temp Calib Failed	Error	-	-	Recalibrate temp
442	Press out of tolerance	Error	Ambient pressure < 15kPa or > 115pKa ➔ error	-	-
443	Boardtemp out of tolerance	Error	Currently in use, only logged	-	GC main board
Out of Tolerance (red_id)					
444	P10V ref out of tolerance	Error	444 to 465 Refvolt out of Tolerance (all voltages on mainboard, part of measuring system) Hazardous by default, basis of all measurement in system	-	Check power supply requirements/ replace main board electronics
445	AIN10V out of tolerance	Error		-	
446	P5V ref out of tolerance	Error		-	
447	P2V5 ref out of tolerance	Error		-	

448	P24V Prim out of tolerance	Error		-	
449	P24V Sec out of tolerance	Error		-	
450	N24V out of tolerance	Error		-	
451	P5V D out of tolerance	Error		-	
452	P5V out of tolerance	Error		-	
453	N5V out of tolerance	Error		-	
454	P15V out of tolerance	Error		-	
455	N15V out of tolerance	Error		-	
456	P5V DAC out of tolerance	Error		-	
457	VREF1 out of tolerance	Error		-	
458	VREF0 out of tolerance	Error		-	
Out of Tolerance (cts_id)					
459	CTS1 out of tolerance	Error	-	Electronics off (all) H2 flow set to 0.0 mL/min	-
460	CTS2 out of tolerance	Error	-		-
461	CTS3 out of tolerance	Error	-		-
462	CTS4 out of tolerance	Error	-		-
463	CTS5 out of tolerance	Error	-		-
464	CTS6 out of tolerance	Error	-		-
465	Tref out of tolerance	Error	-		-
466	Step motor moving	Error	-		-
Option Board Errors					

482	Analogoutput buffer underrun	Alert	Buffer underrun		
483	Analogoutput buffer overflow	Alert	Buffer overrun	Notification Message	-
484	Analogoutput communication error	Alert	-	Notification Message	-
485	Optionboard external events error	Alert	-	Notification Message	-

APPENDIX B: PUBLIC STATEMENT

This public statement is required for the GPL/LGPL licensed software used in this Gas Chromatograph.

GNU General Public License is a free, copy left license for software and other kinds of works.

The 450-GC is using version 2.6.18 of the Linux kernel (provide by Montavista Pro 5.0).

For more details about used GPL/LGPL packages look in the document “License” included on the CD (CP501414) which is included with the 450-GC.