

**Thermo Scientific** 

# TRACE 1300 and TRACE 1310

Gas Chromatographs

**User Guide** 

PN 31715003, Revision F January 2016



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	Strongly Agree	Agree	Neutral	Disagree	Strongly Disagree
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- International Electrotechnical Commission (IEC): 61010-1:2001 (2nd edition) 61010-2-010:2003 (2nd edition) 61010-2-081:2001 (1st edition) + A1:(2003)
- National differences: CAN/CSA C22.2 No. 61010-1 (2nd Edition) UL 61010-1 (2nd Edition)
- EuroNorm (EN): 61010-1:2001 (2nd Edition) 61010-2-010:2004 (2nd Edition) 61010-2-081:2002 (1st Edition)

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- CISPR 11/EN 55011: Group 1 Class A
- IEC/EN 61326-1:2012 (



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# **Preface**

This guide contains detailed information for the use of the TRACE 1300 and TRACE 1310 GC. This guide is organized as follows:

Chapter 1, "Getting Familiar with Your TRACE 1300/TRACE 1310," provides information to familiarize you with your GC including a basic overview of the features and options.

Chapter 2, "TRACE 1310 User Interface," provides instructions for the user interface of your TRACE 1310, including basic information about key functions and menus.

Chapter 3, "TRACE 1300 User Interface," provides instructions for the user interface of your TRACE 1300.

Chapter 4, "Configuration and Method Parameters Setup from CDS," contains the instructions to configure your TRACE 1300/TRACE 1310 and to edit the parameters (according to the front/back injector, front/back detector modules, and optional devices installed), through Xcalibur, Chrom-Card, ChromQuest, or Chromeleon Chromatography Data System.

Chapter 5, "Oven," contains information for controlling and programming the oven.

Chapter 6, "Split/Splitless Injector (SSL) Module," describes the Split/Splitless (SSL) injector module and contains information on controlling and programming the injector setting parameters for the different split/splitless operating modes.

Chapter 7, "Split/Splitless Backflush Injector (SSLBKF) Module," describes the Split/Splitless (SSLBKF) injector module for Backflush applications and contains information on programming the injector in backflush mode.

Chapter 8, "Gas Sampling Valve (GSV) Module," describes the Gas Sampling Valve (GSV) inlet module and provides instructions for programming the inlet parameters.

Chapter 9, "Instant Connect Helium Saver Injector Module (HeS-S/SL)," the Helium Saver (HeS-S/SL) module and provides instructions for programming the inlet parameters.

Chapter 10, "Programmable Temperature Vaporizing Injector (PTV) Module," describes the Programmable Temperature Vaporizing (PTV) injector module and contains information on programming the injector in different operating modes.

Chapter 11, "Programmable Temperature Vaporizing Backflush Injector (PTVBKF) Module," describes the Programmable Temperature Vaporizing (PTVBKF) injector module for Backflush applications and contains information on programming the injector in backflush mode.

Chapter 12, "Flame Ionization Detector (FID) Module," describes the Flame Ionization Detector (FID), and provides instructions for programming the detector parameters and for performing the checkout using different injectors.

Chapter 13, "Nitrogen Phosphorus Detector (NPD) Module," describes the Nitrogen Phosphorus Detector (NPD) module, and provides instructions for programming the detector parameters and for performing the checkout using different injectors.

Chapter 14, "Thermal Conductivity Detector (TCD) Module," describes the Thermal Conductivity Detector (TCD) module, and provides instructions for programming the detector parameter and for performing the checkout using different injectors.

Chapter 15, "Electron Capture Detector (ECD) Module," describes the Electron Capture Detector (ECD) module, and provides instructions for programming the detector parameters and for performing the checkout using different injectors.

Chapter 16, "Flame Photometric Detector (FPD) Module," describes the Flame Photometric Detector (FPD) module, and provides instructions for programming the detector parameters and for performing the checkout using different injectors.

Chapter 17, "Instant Connect Pulsed Discharge Detector (PDD)," describes the Pulsed Discharge Detector (PPD) and provides instructions for programming the detector parameters and for performing the checkout using different injectors.

Chapter 18, "Generic Detector Interface (GDI)," describes the Generic Detector Interface (GDI).

Chapter 19, "Analog Output Interface (AOI) Module," describes the Analog Output Interface (AOI) module.

Chapter 20, "Optional Modules," briefly describes the optional NoVent Microfluidics, FTIR Make-up, and Instant Connect Thermospray SSL Injector modules.

Chapter 21, "Starting Analysis," provides the instructions for running an analysis.

Chapter 22, "Analytical Troubleshooting," contains information to help you diagnose problems with your data. Describes the most common causes of a problem with a baseline, peak, or result, and also provides analytical troubleshooting information for injectors and detectors.

"Glossary," contains definitions of terms used in this guide. It also includes abbreviations, acronyms, metric prefixes, and symbols.

### **About Your System**

Thermo Scientific systems provide high-caliber gas chromatography (GC) instrumentation. Your TRACE 1300/TRACE 1310 GC system can be a stand-alone unit or coupled with other instruments. GC represents a powerful analytical separation technique. Complex mixtures of individual compounds can be injected into the GC, either manually or by using an autosampler, and then separated the eluate for presentation to the detector. The detector generates signals of the GC eluate and its components. These signals are then processed by a Thermo Scientific Chromatography Data System for qualitative identification, as well as accurate and precise quantification of the individual compounds present in the sample.

**IMPORTANT** Thermo Scientific systems are designed to optimize the separation and detection capabilities of GC by providing high performance analytical capabilities for both research and routine applications. More information on the use of this system can be found in related documentation sources and by using the provided contact information.



**WARNING** Thermo Scientific systems operate safely and reliably under carefully controlled environmental conditions. Using the equipment in a manner not specified by the manufacturer might impair the protections provided by the equipment. If you maintain a system outside the specifications listed in this guide, failures of many types, including personal injury or death, might occur. The repair of instrument failures caused by operation in a manner not specified by the manufacturer is specifically excluded from the Standard Warranty and service contract coverage.



**WARNING** Operation of this system requires the use of chemical substances with different hazard specifications. Before using any chemicals, please read the hazard indications and information reported in the Safety Sheet supplied by the manufacturer referring to the relevant CAS (Chemical Abstract Service) number.

### **Power Rating**

TRACE 1300/TRACE 1310 gas chromatograph

- 120 Vac ±10%, 50/60 Hz, 2000 VA
- 230 Vac ±10%, 50/60 Hz, 2000 VA Detailed instrument specifications are in the Product Specifications or Product Brochure.

### **Contacting Us**

Thermo Fisher Scientific provides comprehensive technical assistance worldwide and is dedicated to the quality of our customer relationships and services.

Use http://www.thermoscientific.com address for products information. Use http://www.gc-gcms-customersupport.com/WebPage/Share/Default.aspx address to contact your local Thermo Fisher Scientific office or affiliate GC-GC/MS Customer Support.

## **Related Documentation**

In addition to this guide, Thermo Scientific provides the following documents for the TRACE 1300 and TRACE 1310.

TRACE 1300 and TRACE 1310 Document Set, PN 31715000

- TRACE 1300 and TRACE 1310 Preinstallation Requirements Guide, PN 31715001
- TRACE 1300 and TRACE 1310 Hardware Manual, PN 31715002
- TRACE 1300 and TRACE 1310 User Guide, PN 31715003
- TRACE 1300 and TRACE 1310 Spare Parts Guide, PN 31715004

To suggest ways we can improve the documentation, follow this link to complete our Reader's Survey.

### **Safety Alerts and Important Information**

Make sure you follow the precautionary notices presented in this guide. Safety and other special notices appear in boxes and include the following:

**WARNING** Highlights hazards to humans or the environment. This is the general warning safety symbol and safety alert word to prevent actions that could cause personal injury. Each **WARNING** safety alert is preceded with this safety symbol and another appropriate safety symbol (see "Safety Symbols and Signal Words" on page xxvii) Then it is followed with an appropriate safety precautionary message. When you see a safety alert on your instrument or in the publications, please carefully follow the safety instructions before proceeding.

**CAUTION** Highlights actions that might cause personal injury or instrument damage. We use it to highlight information necessary to prevent personal injury or damage to software, loss of data, invalid test results, or to information that is critical for optimal system performance. A **CAUTION** safety alert is always preceded with an appropriate safety symbol (see "Safety Symbols and Signal Words" on page xxvii). It is followed with an appropriate safety precautionary message. When you see a safety alert on your instrument or in the publications, please carefully follow the safety instructions before proceeding.

**IMPORTANT** Highlights information necessary to prevent damage to software, loss of data, or invalid test results, or it might contain information that is critical for optimal performance of the system.

**Note** Emphasizes important information about a task.

**Tip** Provides information that can make a task easier.

#### **Safety Symbols and Signal Words**

All safety symbols are followed by **WARNING** or **CAUTION**, which indicates the degree of risk of personal injury and/or instrument damage. Cautions and warnings are followed by a descriptor, such as **BURN HAZARD**. A **WARNING** is intended to prevent improper actions that could cause personal injury. A **CAUTION** is intended to prevent improper actions that might cause personal injury and/or instrument damage. You find the following safety symbols on your instrument and/or in this guide:

Symbol	Descriptor
	BIOHAZARD: Indicates that a biohazard <i>will, could</i> , or <i>might</i> occur.
	<b>BURN HAZARD</b> : Alerts you to the presence of a hot surface that <i>could</i> or <i>might</i> cause burn injuries.
A	<b>ELECTRICAL SHOCK HAZARD:</b> Indicates that an electrical shock <i>could</i> or <i>might</i> occur.
	FIRE HAZARD: Indicates a risk of fire or flammability <i>could</i> or <i>might</i> occur.
	<b>EXPLOSION HAZARD</b> . Indicates an explosion hazard. This symbol indicates this risk <i>could</i> or <i>might</i> cause physical injury.
nameet US	<b>FLAMMABLE GAS HAZARD.</b> Alerts you to gases that are compressed, liquefied or dissolved under pressure and can ignite on contact with an ignition source. This symbol indicates this risk <i>could</i> or <i>might</i> cause physical injury.
	<b>GLOVES REQUIRED:</b> Indicates that you must wear gloves when performing a task or physical injury <i>could</i> or <i>might</i> occur.
R	<b>CLOTHING REQUIRED.</b> Indicates that you should wear a work clothing when performing a task or else physical injury <i>could</i> or <i>might</i> occur.
	<b>BOOTS REQUIRED.</b> Indicates that you must wear boots when performing a task or else physical injury <i>could</i> or <i>might</i> occur.
6	<b>MATERIAL AND EYE HAZARD.</b> Indicates you must wear eye protection when performing a task.
$\boldsymbol{\mathbb{A}}$	<b>HAND AND CHEMICAL HAZARD:</b> Indicates that chemical damage or physical injury <i>could</i> or <i>might</i> occur.
×	<b>HARMFUL.</b> Indicates that the presence of harmful material <i>will, could, or might</i> occur.
	<b>INSTRUMENT DAMAGE:</b> Indicates that damage to the instrument or component <i>might</i> occur. This damage might not be covered under the standard warranty.



**LIFTING HAZARD.** Indicates that a physical injury *could* or *might* occur if two or more people do not lift an object.

**MATERIAL AND EYE HAZARD:** Indicates that eye damage *could* or *might* occur.



**READ MANUAL:** Alerts you to carefully read your instrument's documentation to ensure your safety and the instrument's operational ability. Failing to carefully read the documentation *could* or *might* put you at risk for a physical injury.



**TOXIC SUBSTANCES HAZARD:** Indicates that exposure to a toxic substance could occur and that exposure *could* or *might* cause personal injury or death.



**LASER HAZARD.** Indicates that exposure to a laser beam *will, could*, or *might* cause personal injury.



**RADIOACTIVE HAZARD.** Indicates that the presence of radioactive material *could or might* occur.



For the prevention of personal injury, this general warning symbol precedes the **WARNING** safety alert word and meets the ISO 3864-2 standard. In the vocabulary of ANSI Z535 signs, this symbol indicates a possible personal injury hazard exists if the instrument is improperly used or if unsafe actions occur. This symbol and another appropriate safety symbol alerts you to an imminent or potential hazard that *could cause personal injury*.

# **Instrument Markings and Symbols**

The following table explains the symbols used on Thermo Fisher Scientific instruments. Only a few of them are used on the TRACE 1300/TRACE 1310. Those that are used are designated by an asterisk.

	Symbol	Description
		Direct current
*	$\sim$	Alternating current
	$\sim$	Both direct and alternating current
	3~~	Three-phase alternating current
		Earth (ground) terminal
		Protective conductor terminal

**Table 1.** Instrument Marking and Symbols (Sheet 1 of 2)

	Symbol	Description
	, <b> </b> ,	Frame or chassis terminal
	\ ↓	Equipotentiality
*	I	On (supply)
*	$\bigcirc$	Off (supply)
		Equipment protected throughout by DOUBLE INSULATION or REINFORCED INSULATION (Equivalent to Class II of IEC 536)
*		Instruction manual symbol affixed to product. Indicates that the user must refer to the manual for specific <b>WARNING</b> or <b>CAUTION</b> information to avoid personal injury or damage to the product.
	4	Caution, risk of electric shock
*		Caution, hot surface
*		Caution, biohazard
		In-position of a bistable push control
	$\square$	Out-position of a bistable push control
	+	Jack socket
*	X	Symbol in compliance to the Directive 2012/19/EU on Waste Electrical and Electronic Equipment (WEEE) placed on the European market after August, 13, 2005.

#### **Hydrogen Safety Precautions**

Hydrogen is a colorless, odorless, highly flammable gas with the molecular formula  $H_2$  and an atomic weight of 1.00794, making it the lightest element. Hydrogen gas presents a hazard as it is combustible over a wide range of concentrations; at ambient temperature and pressure, this ranges from about 4% to 74.2% by volume.

Hydrogen has a flash point of - 423 °F (- 253 °C) and an auto-ignition temperature of 1,040 °F (560 °C). It has a very low ignition energy and the highest burning velocity of any gas. If hydrogen is allowed to expand rapidly from high pressure, it can self-ignite. Hydrogen burns with a flame that can be invisible in bright light.



**WARNING - EXPLOSION HAZARD** The use of hydrogen as a carrier gas is dangerous. Hydrogen is potentially explosive and must be used with extreme care. Any use of hydrogen gas must be reviewed by appropriate health and safety staff and all installations of hydrogen systems must be performed to applicable codes and standards. Thermo Fisher Scientific assumes no liability for the improper use of hydrogen as a carrier gas.

Before you begin using hydrogen, you should conduct a risk assessment based on the quantity of hydrogen to be used and the conditions of your laboratory. You should ask yourself:

"What hydrogen hazards associated with this project are most likely to occur?"

"What hydrogen hazards associated with this project have the potential to result in the worst consequences?"

- Try to reduce or eliminate the higher risks by using the proper ventilation to remove hydrogen gas before an ignitable concentration can accumulate. You should also consider purging the hydrogen to further reduce hazards and ensure anyone who will be working with hydrogen has basic hydrogen safety training.
- As with laboratory safety in general, be sure to wear safety glasses, laboratory coats, gloves, etc. Typically there are no specific requirements for gaseous hydrogen, other than eye protection when working with a compressed gas. If working with liquid (cryogenic) hydrogen, insulated gloves and protective shoes should be worn in addition to eye protection.
- You should post "No Smoking" and "No Open Flames" signs to identify hydrogen sources and cylinders. Maintain, inspect and leak-test all hydrogen sources regularly.
- All hydrogen shutoff valves should be clearly marked and permanent hydrogen piping should be labeled as such at the supply or discharge point and at regular intervals along its length. Where hydrogen gas piping passes through a wall, the piping should be labeled on both sides of the wall.
- There should also be contingency plans in place should an incident occur.
- The site emergency response team, as well as the local fire department, should know the location of all hydrogen storage tanks.

#### Using Hydrogen with TRACE 1300/TRACE 1310

The use of hydrogen as a carrier gas or as fuel gas for certain flame detectors requires the operator's strict attention and compliance with special precautions due to the hazards involved.

**WARNING - EXPLOSION HAZARD** Hydrogen is a dangerous gas that, when mixed with air, might create an explosive mixture. The use of hydrogen as a carrier gas requires the operator's extreme caution. Special precautions must be taken because of the risk of explosion. The gas chromatograph must be equipped with a hydrogen sensor if you use hydrogen as a carrier gas.



Never use hydrogen as carrier gas in your TRACE 1300/TRACE 1310 system unless your oven has a hydrogen sensor installed. Thermo Fisher Scientific FSEs are not authorized to install or repair any instrument using hydrogen as a carrier gas unless the instrument is equipped with the appropriate sensor.

If your oven does not have a hydrogen sensor already installed, contact your Thermo Fisher Scientific sales representative. To comply with instrument safety requirements, a Thermo Fisher Scientific FSE or authorized service personnel should install the sensor into your TRACE 1300/TRACE 1310.

Hydrogen is a dangerous gas, particularly in an enclosed area when it reaches a concentration corresponding to its lower explosion level (4% in volume). An explosion hazard could develop in the oven when hydrogen is used as a carrier gas if oven elements are not perfectly connected to each other, or if the connection materials are worn out, broken, or otherwise faulty.

Use the following safety precautions when using hydrogen:

- Ensure that all hydrogen cylinders comply with the safety requirements for proper use and storage. Hydrogen cylinders and delivery systems must comply with local regulations.
- Make sure the gas supply is turned completely off when connecting hydrogen lines.
- Perform a leak test to ensure that the hydrogen lines are leak-tight before using the instrument. Repeat this test to eliminate all leaks.
- Ensure your TRACE 1300/TRACE 1310 has a Thermo Fisher Scientific hydrogen sensor installed. A hydrogen sensor continuously monitors the hydrogen level in the oven.

#### **Hydrogen Connection Guidelines**

Use the following guidelines to safely connect hydrogen to your system:

• **Piping**—Hydrogen must be delivered to equipment using appropriate piping and be done in such a way as to pose essentially no hazard to end-users. Piping systems for the delivery of hydrogen should be designed and installed by a person qualified by specific training and experience with hydrogen piping systems.

Stainless steel is usually recommended because it is a safe, cost-effective material. Piping of *black iron* or copper must not be used, as the pipe can become brittle with age. Elastomeric/plastic tubing of various plastics and polymers should not be used, unless the tubing is approved for use with hydrogen. If elastomeric/plastic tubing is used for hydrogen gas delivery, the tubing should be tested for hydrogen permeability to minimize leakage.

The hydrogen piping system must be flexible enough to endure routine thermal expansion and contraction. The system should also include considerations for the most severe condition of temperature and pressure expected during service. Piping and supports must be able to withstand static loading introduced by such things as ice and snow; and dynamic loading from high wind and earthquake.

Caution should be used if burying hydrogen piping. Proper controls should be used to protect against damage and corrosion, and also to prevent Hydrogen from entering a building if there is any leakage.

• Fittings—All fittings must be of the proper type approved or designed for use with hydrogen gas. Use as few fittings as possible to minimize the potential for leaks. After installation, ensure that leak testing is carried out prior to system use, and on a regular basis.

There must be no PTFE tape or other things like *plumber's putty* used to enhance a seal, as this actually is a detriment to a good seal. Ideally the best installation would use stainless steel tubing with appropriate gas-tight fittings.

Welding is usually preferred for joints in hydrogen piping systems since welding provides a better connection and reduces the potential for leaks compared to mechanical fittings. Soft solder joints are not permitted for hydrogen systems (due to the low melting point of soft solder and its potential for brittle failure at cryogenic temperatures). Brazed joints are permitted, but such joints should be protected against the possibility of external fire.

Tubing connections should be clamped to barbed or press-fit type connections. Hose clamps or *jubilee clamps* must not be used.

• Valves—All valves must be suitable for hydrogen service and for the specific operating conditions. Valves, including regulators, must not be used for hydrogen, unless they are designed and identified for such a use. Ball valves are often chosen because of their superior leak tightness through the valve seat. Pneumatic operators are usually chosen for remotely operated valves so that potential ignition sources (electricity) are remote from the valve.

Manual shutoff valves should be provided near each point of use, within immediate reach. If a hydrogen cylinder or hydrogen generation system is located within immediate reach, a separate point-of-use shutoff valve is usually not necessary.

Line regulators that have their source away from the point of use should have a manual shutoff valve near the point of use.

An emergency gas shutoff device in an accessible location outside the use area should be provided in addition to the manual point-of-use valve in each educational and instructional laboratory space that has a piped gas supply system.

If necessary, the piping system should have uninterruptible pressure relief. The pressure relief system should be designed to provide a discharge rate sufficient to avoid further pressure increase and should vent to a safe location outside or to a ventilation system exhaust.

#### **Purchasing Hydrogen**

Use the following guidelines when purchasing hydrogen:

• Hydrogen Generator—Because it minimizes the amount of hydrogen present and reduces the degree of hazard, a hydrogen generator (also called an electrolyzer) is the safest way to purchase hydrogen in the quantity used in gas chromatography/mass spectroscopy systems.

However, to minimize the degree of hazard, operate the hydrogen generator only in a non-explosive environment because hydrogen buildup can be ignitable. Thus, your ventilation system for the room or lab hood where the hydrogen generator operates must maintain an air exchange rate at least two orders of magnitude greater than the maximum hydrogen production rate of the hydrogen generator. Follow the manufacturers' directions about proper use and maintenance of the regulator.

To prevent the possibility of releasing hydrogen, set the hydrogen generator to shut down if:

- There is a loss of flow to the ventilation system
- A hydrogen detector alarms at 25% of the lower flammable limit of hydrogen in air.

Vent the oxygen exhausted by the electrolyzer to the outside as well.

• Hydrogen Cylinder—Hydrogen can be delivered in standard laboratory gas bottles or cylinders. These cylinders have a limited amount of hydrogen in them and are a safe way to transport and store hydrogen. Always secure, compressed hydrogen gas cylinders, like all compressed gas cylinders, in an upright position, ideally with a non-combustible chain or cable. If the cylinder falls over, the valve can fall off, causing the pressurized cylinder to take off like a rocket, leading to the release of hydrogen and possibly an explosion, severe injury, or death. Never crack a hydrogen cylinder valve to remove dust or dirt from fittings prior to attaching a regulator, as there is a risk of self-ignition.

#### **Properly Storing Hydrogen**

Storing and handling compressed hydrogen gas and cryogenic liquid hydrogen present potential health and safety hazards. Using proper storage and handling techniques is essential to maintaining a safe work environment. Use the following guidelines when storing hydrogen:

- Store spare hydrogen gas cylinders outside and away from doors, windows, building air intake vents, structures, and vehicle routes. This precaution applies when the hydrogen is or is not in use. Indoor storage of spare hydrogen cylinders has special requirements, which are beyond the scope of this document. Documentation for each vessel should include a description of the vessel, a list of available drawings or other documents, the most recent inspection results, and the responsible person's name.
- Prevent spare cylinders from toppling by wrapping them with chains. The chains should also be protected against corrosion and excessive heat.
- Separate spare hydrogen cylinders from oxidizing gases (such as oxygen) with a 5 ft. (1.5 m) tall fire barrier with a half-hour fire rating or place the cylinders at least 20 ft. (6 m) apart.
- When moving hydrogen cylinders:
  - Remove the regulator and replace the cylinder valve cap before moving.
  - Move cylinders on cylinder carts or with other appropriate transport devices.
  - Never roll or drop a cylinder and never lift a cylinder by its protective cap.
- Bulk hydrogen systems include either gaseous or liquid hydrogen in fixed installations; in some gas systems a semi-permanent trailer (tube trailer) can be used. Storage vessels for compressed hydrogen gas or liquid hydrogen should be designed, constructed, tested, and maintained in accordance with applicable codes and standards. Bulk hydrogen systems represent a level of complexity again which is beyond the scope of this document; however some general guidelines are provided.
- The bulk hydrogen storage system should not be located beneath electric power lines, close to other flammable gases/liquids, or close to public areas. It should be readily accessible to authorized personnel and delivery equipment, but protected from physical damage or tampering.
- As liquid hydrogen systems also have a cryogenic hazard, additional safety considerations for the use of cryogenic liquids might be necessary.

#### Hydrogen Safety Codes, Standards and References

The following list of safety codes, standards, and references is in no way an exhaustive list. In fact, there may be federal, state, or local codes that apply to your specific location. Check with all appropriate agencies with jurisdiction before installing or using a hydrogen system.

- Air Products Safetygram #4 Gaseous Hydrogen
- ANSI/AIAA standard for hydrogen safety guidelines is AIAA G-095-2004, Guide to Safety of Hydrogen and Hydrogen Systems
- ASME B31.1, Power Piping Code

- ASME B31.3, Process Piping Code
- ASME B31.8, Gas Transmission and Distribution Systems
- BCGA Code Of Practice CP4 Industrial Gas Cylinder Manifolds and Gas Distribution
   Pipework
- BCGA Code Of Practice CP33 The Bulk Storage of Gaseous Hydrogen at Users' Premises
- CGA G-5, Hydrogen
- CGA G-5.4, Standard for Hydrogen Piping Systems at Consumer Locations
- CGA G-5.5, Hydrogen Vent Systems
- CGA G-5.6, Hydrogen Pipeline Systems
- CGA G-5.8, High Pressure Hydrogen Piping Systems at Consumer Locations.
- FM Global Property Loss Prevention Data Sheets 7-50: Compressed Gases in Cylinders
- FM Global Property Loss Prevention Data Sheets 7-91: Hydrogen
- IGC Doc 121/04/E, Hydrogen Transportation Pipelines System Design Features
- NASA
- NSS 1740.16 Safety Standard For Hydrogen And Hydrogen Systems Guidelines for Hydrogen System Design, Materials Selection, Operations, Storage, and Transportation
- NFPA 52, Vehicular Fuel Systems Code
- NFPA 55, Standard for the Storage, Use, and Handling of Compressed Gases and Cryogenic Fluids in Portable and Stationary Containers, Cylinders, and Tanks, 2005 Edition
- NFPA 68, Standard on Explosion Protection by Deflagration Venting
- NFPA 70, National Electrical Code
- NFPA 497, Recommended Practice for the Classification of Flammable Liquids, Gases, or Vapors and of Hazardous (Classified) Locations for Electrical Installations in Chemical Process Areas
- NFPA 13, Standard for the Installation of Sprinkler Systems
- NFPA 45, Standard on Fire Protection for Laboratories Using Chemicals
- NFPA 55, Standard for the Storage, Use, and Handling of Compressed Gases and Cryogenic Fluids in Portable and Stationary Containers, Cylinders, and Tanks
- NFPA 68, 2007 Standard on Explosion Protection by Deflagration Venting
- NFPA 69, Standard on Explosion Prevention Systems

- NFPA 91, Standard for Exhaust Systems for Air Conveying of Vapors
- NFPA 255, Standard Method of Test of Surface Burning Characteristics of Building Materials
- OSHA 29CFR1910.103 1910.103 Hydrogen

### **Hazardous Substances Precautions**



**WARNING** Before using hazardous substances (toxic, harmful, etc.), please read the hazard indications and information reported in the applicable Material Safety Data Sheet. Use Personal protection according to the safety requirements.

#### **Venting Toxic Gases**

When analyzing toxic compounds be aware that during the normal operation of the GC some of the sample may be vented outside the instrument through the inlet and detector exits; therefore the exhaust gases must be vented to a fume hood. Consult local Environmental and Safety Regulations for instructions in exhausting fumes from your system.

# **Liquid Nitrogen Safety Precautions**

Liquid nitrogen is a colorless, odorless, extremely cold liquid and gas under pressure. It can cause rapid suffocation when concentrations are sufficient to reduce oxygen levels below 19.5%. A Self Contained Breathing Apparatus (SCBA) may be required. Contact with liquid or cold vapors can cause severe frostbite. Cold vapors in the air will appear as a white fog due to condensation of moisture. Oxygen concentrations must be monitored in the release area. All cryogenic liquids produce large volumes of gas when they vaporize.

**WARNING** Before using Liquid Nitrogen, read the hazard indications and the instructions reported in the Safety sheet supplied by the manufacturer with reference to the CAS number (Chemical Abstract Service) 7727-37-9.



Use personal protection:

- Protective gloves: Loose fitting thermal-insulated or leather gloves.
- Eye protection: Full face shield and safety glasses are recommended.
- **Other protective equipment**: Safety shoes when handling containers. Long sleeved shirts and trousers without cuffs. Work clothing that sufficiently prevents skin contact should be worn.

# **Carbon Dioxide Safety Precautions**

Carbon dioxide is a colorless, cryogenic liquid. At low concentrations, is odorless. At higher concentrations carbon dioxide will have a sharp, acidic odor. At concentrations between 2 and 10%, Carbon dioxide can cause nausea, dizziness, headache, mental confusion, increased blood pressure, and increased respiratory rate. If the gas concentration reaches 10% or more, suffocation and death can occur within minutes. Contact with the cold gas can cause freezing of exposed tissue. Moisture in the air could lead to the formation of carbonic acid that can be irritating to the eyes. All forms of carbon dioxide are noncombustible. Carbon dioxide is heavier than air and should not be allowed to accumulate in low lying areas.

**WARNING** Before using carbon dioxide, read the indications of hazard and the instructions reported in the Safety sheet supplied by the manufacturer with reference to the CAS number (Chemical Abstract Service) 124-38-9.

Use personal protection:

- Protective gloves: Loose fitting thermal insulated or leather gloves.
- Eye protection: Full face shield and safety glasses are recommended.
- **Other protective equipment**: Safety shoes when handling containers. Long sleeved shirts and trousers without cuffs. Work clothing that sufficiently prevents skin contact should be worn.



# **Getting Familiar with Your TRACE 1300/TRACE 1310**

This chapter provides information to familiarize you with the TRACE 1300 and TRACE 1310 gas chromatographs.

#### Contents

- Instrument Basics
- Labels Location on the Instrument
- Oven
- Injector Modules Housing
- Detector Modules Housing
- External Modules Housing
- Gas Inlet Ports
- Electronic Module
- TRACE 1310 User Interface
- TRACE 1300 User Interface

# **Instrument Basics**

The TRACE 1300/TRACE 1310 comprises:

- The **injector**, where the sample is introduced. The following injectors modules are available:
  - Split/Splitless Injector (SSL)
  - Split/Splitless Injector for Backflush applications (SSLBKF)
  - Gas Sampling Valve (GSV)
  - Helium Saver S/SL (HeS-S/SL)
  - Programmable Temperature Vaporizing Injector (PTV)
  - Programmable Temperature Vaporizing Injector for Backflush applications (PTVBKF)
- The **oven**, where the analytical column is installed to perform the sample separation into its compounds.
- The **detector**, where the separated sample compounds are detected. The following detectors modules are available:
  - Flame Ionization Detector (FID)
  - Electron Capture Detector (ECD)
  - Nitrogen Phosphorus Detector (NPD)
  - Thermal Conductivity Detector (TCD)
  - Flame Photometric Detector (FPD)
  - Pulsed Discharge Detector (PDD)
  - Up to two mass spectrometers (MS) can be coupled to a GC version for MS.
  - A Generic Detector Interface (GDI) for third-party detectors.
- The electronic module for the power supply of the instrument at 120 Vac or 230 Vac.
- Two housings to accommodate **external modules** as Auxiliary Temperature/Cryo, Auxiliary Gas, NPD thermionic source power supply, GDI Electrical Interface (part of the Generic Detector Interface), and Analog Output Interface.
- The touch screen as a **user interface** for TRACE 1310.
- The status panel as a **user interface** for TRACE 1300.

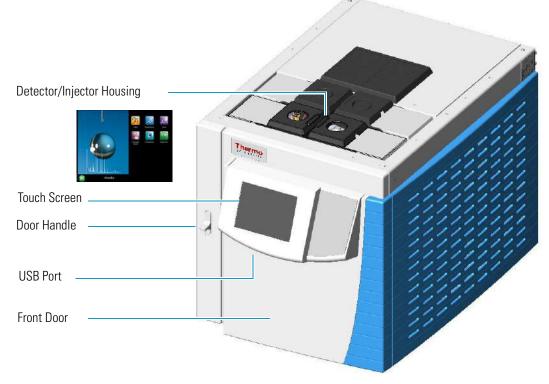
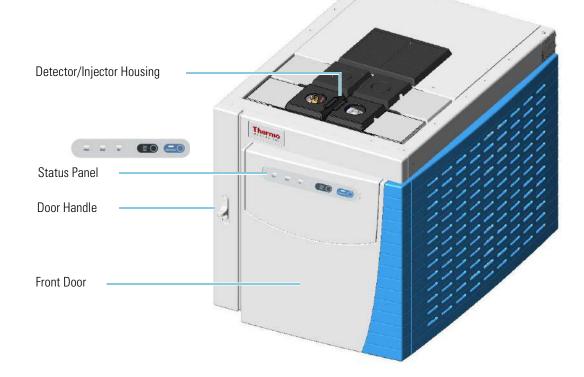


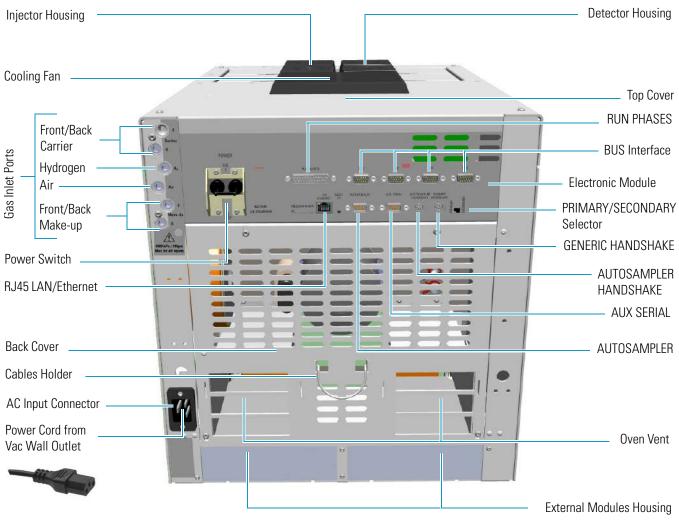
Figure 1. TRACE 1310 Gas Chromatograph with Touch Screen

Figure 2. TRACE 1300 Gas Chromatograph with Status Panel



Instrument Basics





**Note** The TRACE 1300/TRACE 1310 can be furnished factory coupled with the TRACE 1310 Auxiliary Oven. See Figure 4.



The TRACE 1310 Auxiliary Oven is described into a dedicated Instruction Manual (*TRACE 1310 Auxiliary Oven Instruction Manual PN 31715011*) as additional section of the *TRACE 1300 and TRACE 1310 User Guide* and *TRACE 1300 and TRACE 1310 Hardware Manual*.

# Labels Location on the Instrument

The following illustrations show the location of the labels attached on the instrument.

Figure 5. TRACE 1300 and TRACE 1310: Serial Number and US Patents Labels

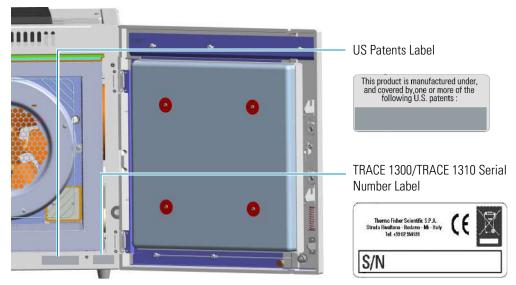


Figure 6. Electronic Module: Serial Number, Electrical Data, MAC Code and LAN Data



Electronic Module Serial Number Label and Electrical Data

Thermo Fisher Scientific S.p.A.	Thermo Fisher Scientific S.p.A.	
Strada Rivoltana - Rodano - Mi - Italy	Strada Rivoltana - Rodano - Mi - Italy	
Tel. +39 02 95051	Tel. +39 02 95051	
Model:	Model:	
Trace 1300 series CE	Trace 1300series CE	
S/N:	S/N:	
Code: 432.101.68	Code: 432.101.67	
120V ~ 2000 VA 50/60 Hz	230V~ 2000 VA 50/60 Hz	

Power Supply 120 Vac or 230 Vac Alert Label



MAC Code and LAN Data Label

MAC: XX-XX-XX-XX-XX-XX
IP:

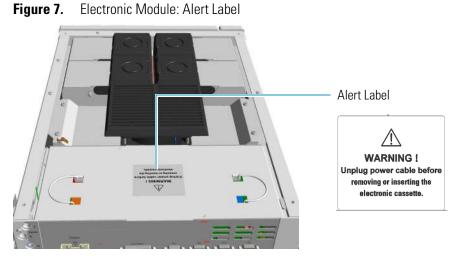
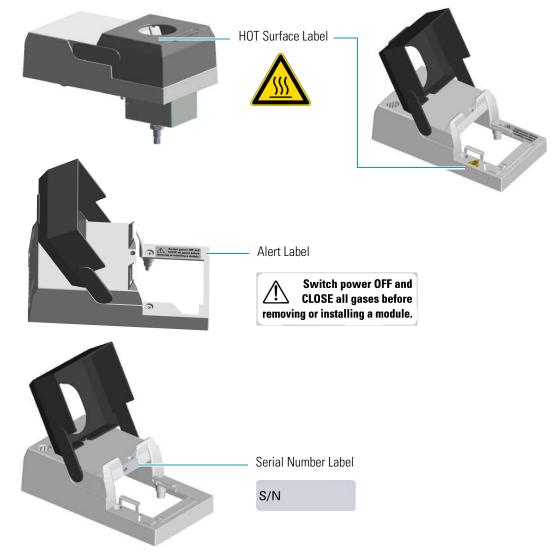
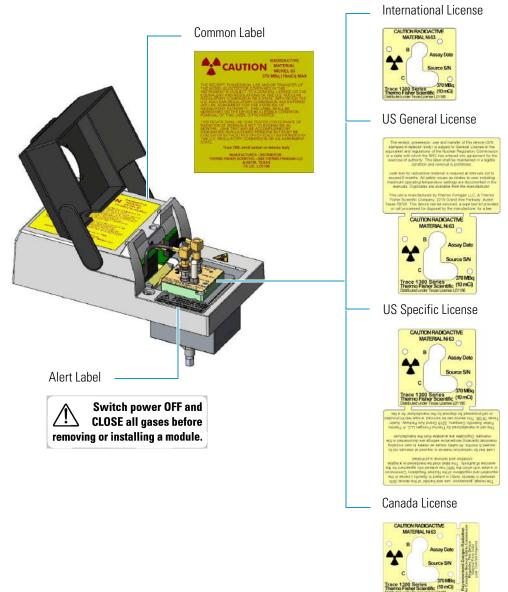


Figure 8. Injector/Detector Modules: Hot Surface and Alert Labels



#### Figure 9. ECD Detector Module Labels

Caution Radioactive Material Labels according to International, USA, and Canada Licenses



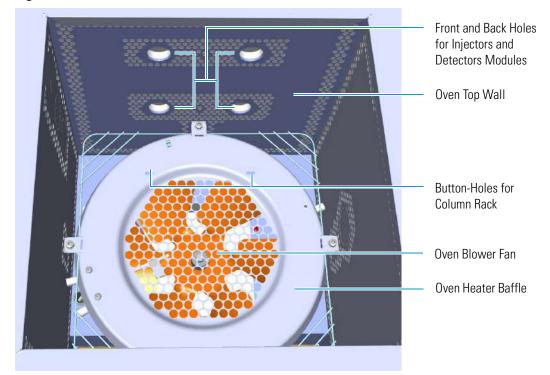
**IMPORTANT** Exclusively for ECD detector module used in **China**, the "Caution Radioactive Material Ni63" labels are replaced by the followings:





# Oven

To access the oven, open the GC front door by pushing down the door handle on the left. See Figure 10.





If the GC is powered on, opening the front door activates a magnetic safety switch, which automatically powers off the oven's heater, the motor of the air circulation fan, and the cryogenic system when installed.

The oven has the following capabilities:

- Maximum temperature increase rate of 125°C/min., 32 linear temperature ramps and 33 plateaus, and a minimum operating temperature of a few degrees above ambient, which is obtained by two modulated cooling flaps controlled by the GC.
- Maximum temperature of 450 °C.

With a cryogenic option installed, the oven temperature can reach -50 °C with carbon dioxide or -100 °C with liquid nitrogen. The coolant flow is controlled by an optional cryogenic system. The Oven cryogenic systems with carbon dioxide and with liquid nitrogen are shown in Figure 11 and Figure 12 respectively.

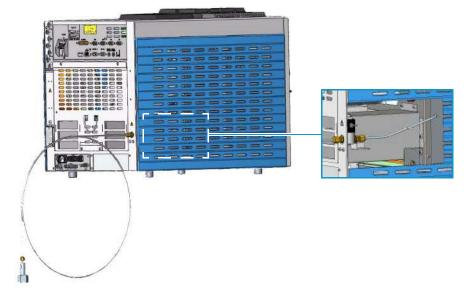


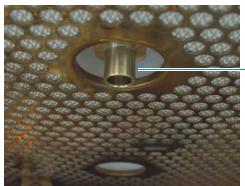
Figure 11. Oven Cryo System with Carbon Dioxide

Figure 12. Oven Cryo System with Liquid Nitrogen



### **Column Fittings**

The fittings for the connection of the column to the front/back injector and detector modules protrude into the oven through the front/back holes on the oven top wall. See Figure 13.



**Figure 13.** Column Fitting Protruding

Fitting for the connection of the column

### **Ducts for MS Connection**

The ducts prearranged on the walls of the oven box allow the coupling between the GC and a Thermo Scientific ISQ, TSQ 8000 Series, DSQ II, ITQ, and TSQ Quantum mass spectrometers (MS).

When connecting the mass spectrometer, introduce its transfer line inner tube into the GC oven through the proper duct. For further details, please refer to **Installation** section in the *TRACE 1300 and TRACE 1310 Hardware Manual*.

Figure 14 shows the position of the ducts on the left side wall through which the relevant ISQ, TSQ 8000 Series, DSQ II, and ITQ transfer line inner tube will protrude into the GC oven.

Figure 14. Duct for the Coupling with an ISQ, TSQ 8000 Series, DSQ II, or ITQ Mass Spectrometer

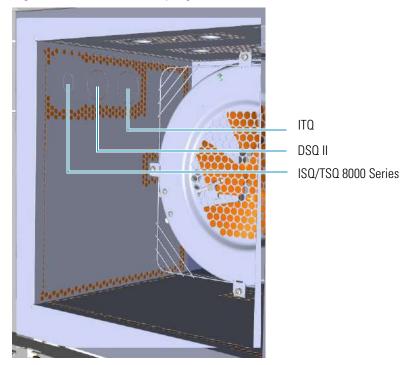


Figure 15 shows the position of the duct on the right side wall through which the TSQ Quantum transfer line inner tube will protrude into the GC oven.

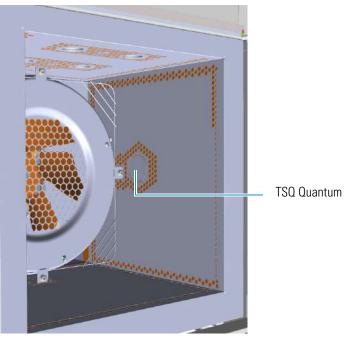


Figure 15. Ducts for the Coupling with a TSQ Quantum Mass Spectrometer

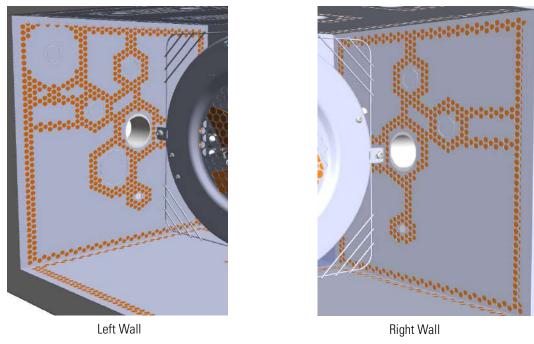
You can also configure a stand-alone GC for coupling with an ISQ, TSQ 8000 Series, DSQ II, ITQ, or TSQ Quantum mass spectrometer by installing the dedicated upgrade kit. For further details, please refer to the section *Upgrading a Stand Alone TRACE 1300/TRACE 1310 to MS Version* in the *TRACE 1300 and TRACE 1310 Hardware Manual*.

**Note** The coupling of the GC with a Thermo Scientific high resolution mass spectrometer (HRMS) requires a dedicated oven box with specific wall ducts. See the section "Ducts for HRMS Connection" on page 13.

### **Ducts for HRMS Connection**

The ducts prearranged on the walls of the dedicated oven box allow the coupling of the GC and a Thermo Scientific HRMS such as DFS, IRMS, and ICP-MS. See Figure 16.



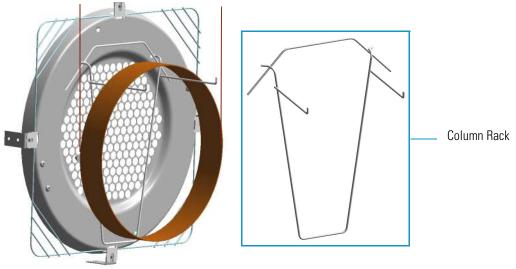


Properly connect the mass spectrometer, introducing the transfer line inner tube into the GC oven through the proper duct. For further details, please refer to the **Installation** section in the *TRACE 1300 and TRACE 1310 Hardware Manual*.

### **Column Rack**

Two analytical capillary columns can be installed into the oven in front and/or back position by using the column rack as their support. See Figure 17.





Install the column rack by inserting the two hooks into the corresponding slots on the oven heater baffle. See Figure 18.

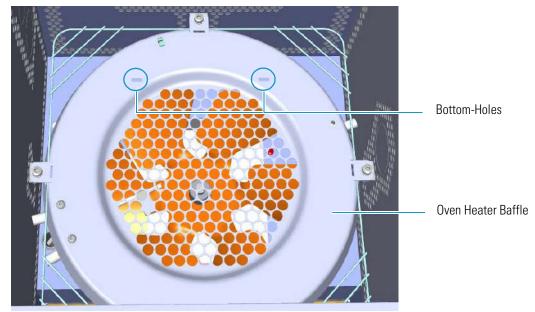


Figure 18. Column Rack (2)

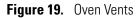
For further details, please refer to the **Installation** section of the *TRACE 1300 and TRACE 1310 Hardware Manual*.

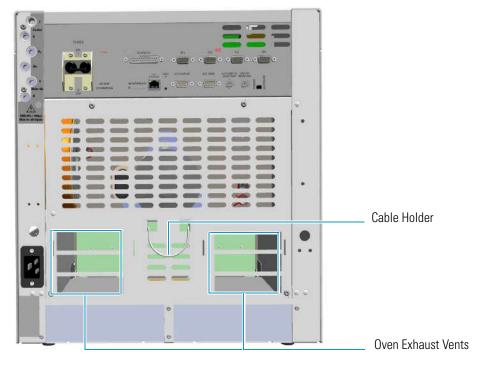
#### **Oven Vents**

The oven vents at the back of the GC discharge hot air up to 450 °C (842 °F) during cooling.



**WARNING** Oven exhaust can cause severe burns. Avoid working behind the instrument oven vents during cool-down cycles. There should be at least 30 cm (12 in.) free space behind the instrument to allow the exhaust to dissipate. Do not expose gas tanks or bottles, chemicals, regulators, electrical cables, or other temperature-sensitive items to oven exhaust.





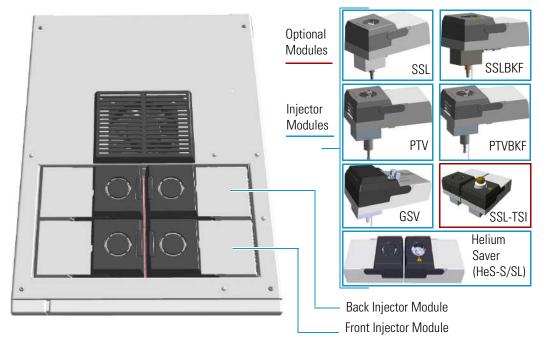


**CAUTION** To avoid contact with the hot air from the vents, gather the electrical cables into the cables holder.

# **Injector Modules Housing**

Your TRACE 1300/TRACE 1310 accommodates up to two injector modules in the **front/back** sites located on the **right** side. See Figure 20.

The housing must always be occupied by both the modules. According to the configuration of your TRACE 1300 or TRACE 1310 GC, a dummy module is present in the free site where the injector module is not installed.





Each front/back position has its own electrical and gas connections as shown in Figure 21.

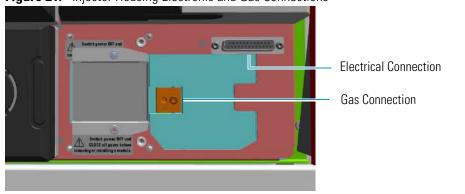


Figure 21. Injector Housing Electronic and Gas Connections

**Note** Where a dummy module is installed, the gas connection is blocked by a plug.

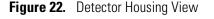
When the injector module is correctly installed and fixed into its own site, the fitting for the column connection protrudes into the interior of the oven through the top wall.

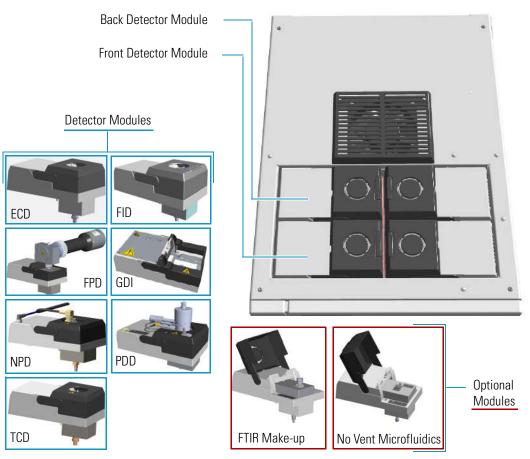
For details about the **Injector** modules see the following chapters:

- Chapter 6, "Split/Splitless Injector (SSL) Module," on page 139
- Chapter 7, "Split/Splitless Backflush Injector (SSLBKF) Module," on page 153
- Chapter 8, "Gas Sampling Valve (GSV) Module," on page 165
- Chapter 9, "Instant Connect Helium Saver Injector Module (HeS-S/SL)," on page 175
- Chapter 10, "Programmable Temperature Vaporizing Injector (PTV) Module," on page 193
- Chapter 11, "Programmable Temperature Vaporizing Backflush Injector (PTVBKF) Module," on page 217

# **Detector Modules Housing**

Your TRACE 1300/TRACE 1310 accommodates up to two detector modules in the **front/back** sites located on the **left** side. An optional module can be installed instead of a detector module. See Figure 22. The housing must always be occupied by both the modules. According to the configuration of your TRACE 1300 or TRACE 1310 GC, a dummy module is present in the free site where the detector module is not installed.





Each front/back position has its own electrical and gas connections as shown in Figure 23.

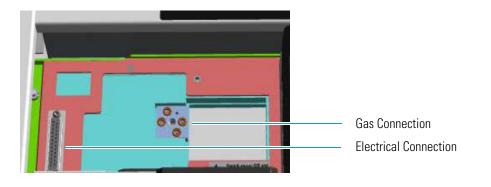


Figure 23. Detector Housing Electronic and Gas Connections

**Note** Where a dummy module is installed, the gas connection is blocked by a plug.

When the detector module is correctly installed and fixed into its own site, the fitting for the column connection protrudes into the interior of the oven through the top wall.



**IMPORTANT** When the GC is equipped with a FID, ECD, NPD, TCD, or PDD detector module and a FPD detector module, this last should be installed into the **back** site.

For details about the **Detector** modules see the following chapters:

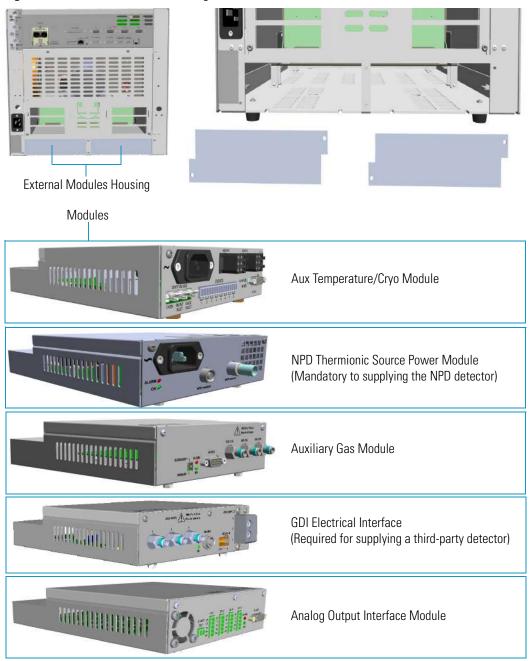
- Chapter 12, "Flame Ionization Detector (FID) Module," on page 233
- Chapter 13, "Nitrogen Phosphorus Detector (NPD) Module," on page 241
- Chapter 14, "Thermal Conductivity Detector (TCD) Module," on page 251
- Chapter 15, "Electron Capture Detector (ECD) Module," on page 261
- Chapter 16, "Flame Photometric Detector (FPD) Module," on page 271
- Chapter 17, "Instant Connect Pulsed Discharge Detector (PDD)," on page 285
- Chapter 18, "Generic Detector Interface (GDI)," on page 295

**Note** For details about the No Vent Microfluidics and FTIR Make-up **optional modules**, see the Chapter 20, "Optional Modules," on page 307.

# **External Modules Housing**

Your TRACE 1300/TRACE 1310 can accommodate up to two external modules in the appropriate sites located in the back lower part of the GC. See Figure 24.

Figure 24. External Modules Housing



• Aux Temperature/Cryo Module — The module controls the transfer line heater (Aux temperature) for two mass spectrometers, the solenoid valve for the Oven cryogenic system, the solenoid valves for the front/back PTV/PTVBKF cryogenic systems, and up to eight solenoid valves (24 V, 2 W max) for the activation of external events.

**Note** In case of the **GC Transfer Line**, both the **HEATER 1** and **HEATER 2** connectors on the Aux Temperature/Cryo Module are used for controlling the heaters of the GC Transfer Line. Note the Y-shape cable in Figure 25.

#### Figure 25. GC Transfer Line



The GC Transfer Line uses line voltage to power two heated zones. Each zone has two cartridge heaters and one RTD mounted within an aluminum heat distributor covering the stainless transfer line.

The first heated zone connected to HEATER 1 on the auxiliary temperature module heats the section of column closest to the ion source of the mass spectrometer.

The second heated zone connected to HEATER 2 on the auxiliary temperature module heats the section of column closest to the GC. Each temperature zone is settable up to 400 °C. Because the zones are physically connected to the same transfer line each zone will supply some heat to the other and their set point should be the same or within approximately 50 °C in order to maintain stable temperature control.

The GC Transfer Line covers the nut of the MS transfer line during normal operation and should be maintained at a constant temperature to avoid leaks do to expansion and contraction of the seal between atmosphere and vacuum. When the GC Transfer Line temperature must be reduced, the MS should be checked for vacuum leaks and the ferrule should be re-tightened accordingly. To avoid leaks into the MS, it is not recommended to set the transfer line above 280°C when using graphite Vespel<sup>®</sup> ferrules on the MS transfer line as these ferrules eventually develop vacuum leaks at high temperature. Because the pressure at the vacuum end of the column is at reduced pressure, most compounds up to hydrocarbons such as C44 will elute at 280 °C or less. Metal ferrules may be used if higher temperatures are required.

- NPD Thermionic Source Power Module The module controls the power supply and the signals of the thermionic source of the NPD detector module.
- Auxiliary Gas Module The module controls the pressure of up to three auxiliary carrier gases.
- GDI Electrical Interface With the GDI Mechanical Module it forms the Generic Detector Interface (GDI) for the installation of a third-party detector. This interface controls the pressure and the flow up to three detector gases, the temperature of the third-part detector, and the acquisition of the analog signal from the third-part detector. For further details see Chapter 18, "Generic Detector Interface (GDI)," on page 295.
- Analog Output Interface The module receives digital signal values from up to four detectors and convert them to analogue signals. For further details see Chapter 19, "Analog Output Interface (AOI) Module," on page 303.

## **Gas Inlet Ports**

You can find the gas inlet ports on the back upper left corner of the instrument. They are directly connected to the injector and detector housing and supply the relevant front/back modules. See Figure 26.

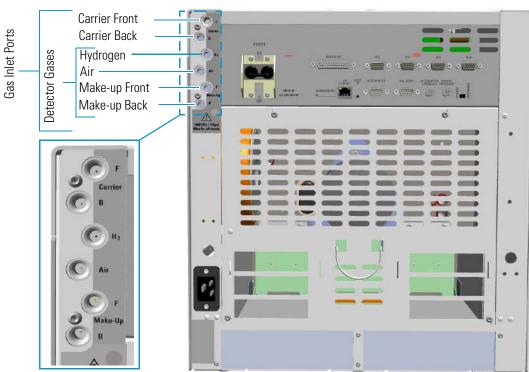


Figure 26. Gases Inlet Connections

**Note** Do not connect any gases to the GC other than those referenced in the manual.



**CAUTION** The maximum nominal inlet pressure for all the inputs is 1000 kPa (145 psig; 10 bar), as indicated on the label under the gas inlets ports on the back of the GC. The working inlet pressure range is from 400 kPa (58 psig; 4 bar) to 1000 kPa (145 psig; 10 bar).

The gas recommendations for capillary and wide-bore column systems are detailed in Table 1:

Table 1. Capil	ary and Wide-	Bore Column	Gas Recommendations
----------------	---------------	-------------	---------------------

Detector type	Carrier gas	Fuel gas	Make-up gas
FID	Helium, Nitrogen, Hydrogen	Hydrogen + Air	Nitrogen
NPD	Helium, Nitrogen, Hydrogen	Hydrogen + Air	Helium, Nitrogen
ECD	Helium, Nitrogen, Argon	None	Nitrogen, Argon/5% Methane
TCD	Helium, Nitrogen, Hydrogen, Argon	None	Same as carrier
FPD	Helium, Hydrogen	Hydrogen + Air	None
PDD	Dedicated Helium supply as discharge ga	S	

**WARNING** Before using gases, carefully read the hazard indications and information reported in the Safety Sheet supplied by the manufacturer referring to the CAS (Chemical Abstract Service) number. It is the user's responsibility to see that all local safety regulations for the use of gases are obeyed.



All Thermo Scientific gas chromatographs normally use an inert gas as carrier gas. If you wish to use hydrogen as carrier gas, you must install a hydrogen sensor. Contact a Thermo Fisher Scientific sales representative if you plan to use hydrogen as the carrier gas in your new TRACE 1300/TRACE 1310. If you do not have a hydrogen sensor, you **must** use an inert carrier gas.

Thermo Fisher Scientific FSEs are not authorized to install or repair any instrument using hydrogen as a carrier gas unless the instrument is equipped with the appropriate sensor.

**WARNING** For precautions and use of hydrogen with the TRACE 1300/TRACE 1310, see the section "Hydrogen Safety Precautions" on page xxix for details.

### **Using a Hydrogen Sensor**

The hydrogen sensor must be installed into the back of the GC as shown in Figure 27.

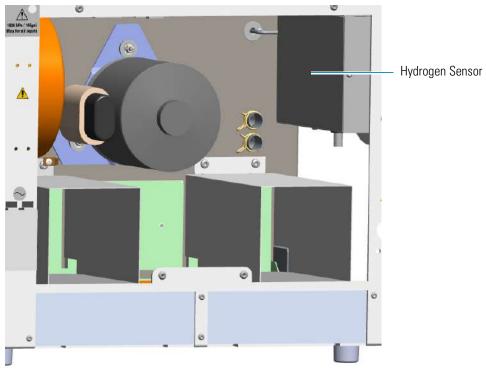


Figure 27. Hydrogen Sensor Housing

To install the hydrogen sensor into the GC, please refer to the *TRACE 1300 and TRACE 1310 Hardware Manual*.

The reliability of the sensor depends on careful maintenance. When the sensor is in use, you must periodically check its operating performance and calibration as recommended by the manufacturer. Refer to your hydrogen sensor's instruction manual for maintenance guidelines.

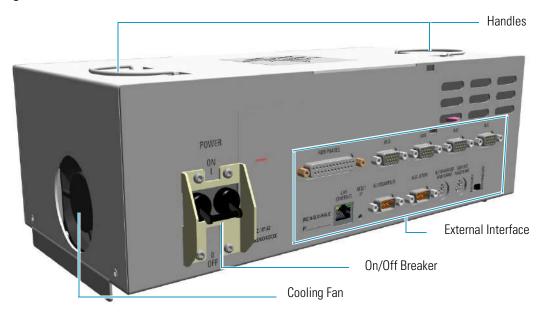
# **Electronic Module**

The Electronic Module contains the power and electronic circuits that operate and control the instrument. A proper module is installed according to 120 Vac or 230 Vac power supply.



**WARNING** This operation must be carried out only by Thermo Fisher Scientific technical personnel. The Electronic Module must be removed only when necessary. Before accessing to the Electronic Module, turn off the power supply to the instrument and unplug the power cord.

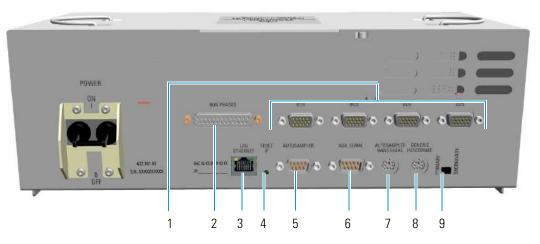
Figure 28. Electronic Module



The Electronic Module (see Figure 28) includes:

- The **Power Switch** (breaker 16 A for 120 Vac; 10 A for 230 Vac power supply) marked **Power** to power the instrument On/Off.
  - Position I = instrument On
  - Position **O** = instrument Off
- Two handles on the top to allow an easy upwards extraction of the module from the GC.
- A cooling **fan** on the left-hand side allows cool-air circulation into the module.

• The **external interface** includes the connectors to make electrical connections and communication among the TRACE 1300/TRACE 1310 and the external devices such an autosampler or a computer. Figure 29 shows the components present on the external interface.



#### Figure 29. Electronic Module: External Interface

- 1. Four 15-pin female connectors marked **Bus** for communication with external devices.
- 2. A 25-pin female connector marked **Run Phases** for connect to external devices such as a cold trap.
- 3. A RJ45 connector marked **LAN/Ethernet** for the network connection of the TRACE 1300/TRACE 1310.
- 4. A button marked Reset IP to reset the IP address.
- 5. A 9-pin male connector marked **Autosampler** to dialog with the autosampler.
- 6. A 9-pin male connector marked **Aux Serial** to dialog with a second autosampler or with future application external devices.
- 7. A 6-pin connector marked **Autosampler Handshake** to synchronize the TRACE 1300/TRACE 1310 with the autosampler.
- 8. An 8-pin connector marked **Generic Handshake** to synchronize the TRACE 1300/TRACE 1310 with external devices.
- 9. A selector marked **Primary/Secondary** to select a primary and a secondary gas chromatograph when the TRACE 1300/TRACE 1310 is coupled with another GC.

#### **Function of the Electronic Boards**

The following information details the function of each electronic board inside the Electronic Module:

- Oven CPU and Power Control (OVN-HRM) This board interlocks with the Electronic Module and the rest of the system through the **Backplane board** (BKP-HRM). The following operations are performed:
  - Receives the main voltage through the power switch On/Off (breaker).
  - Furnishes the main voltage to the main transformer.
  - Generates voltages to supply the electronic boards, injector and detector modules, motors, fans, and oven heater.
  - Includes the power selector 120/230 Vac and 2 VA flat compact transformer furnishing 12 V to the main CPU board.

Five protecting fuses are present. See Table 2.

Table 2.Protecting Fuses

Fuse	Type for 120 Vac	Type for 230 Vac	Protections
F1	T10A; IEC 127/III	T6.3A; IEC 127/III	Toroidal main
F2	- (5 x 20 mm)	(5 x 20 mm)	transformer
F3	T200mA; IEC 127/III	T100mA; IEC 127/III	Internal flat compact
F4	- (5 x 20 mm)	(5 x 20 mm)	transformer
F5	T2A; IEC 127/III (5 x 20 mm)	T1A; IEC 127/III (5 x 20 mm)	Fan motor

• Main CPU (CPU-HRM) — This board controls all the instrument operations. Actuates the safety cut-off device, which puts the instrument in safe condition, when an alarm occurs.

**IMPORTANT** A Li 3V; 220 mA/h unrechargeable battery is present. The battery replacement must be performed by specialized Thermo Fisher Scientific technical personnel.

• External Interface board (EXT-HRM) — This board includes the Run Phases and Bus connectors for the communication with external devices.

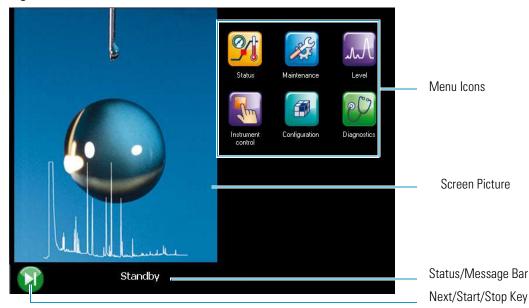
# **TRACE 1310 User Interface**

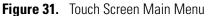
The TRACE 1310 user interface is a **touch screen** that provides immediate and detailed status displays, and guidance for ease of use and local control. See Figure 30.



Figure 30. TRACE 1310 User Interface

When the TRACE 1310 is powered on, the main menu appears as shown in Figure 31.





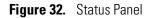
Each instrument's function is associated with an icon that opens the relevant menu: Status, Maintenance, Level, Instrument Control, Configuration, and Diagnostic.

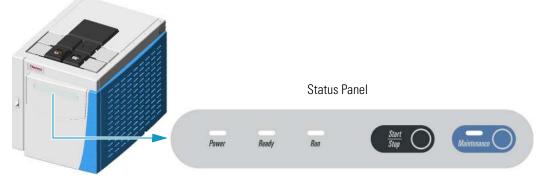
See Chapter 2, "TRACE 1310 User Interface." for details. To operate with the TRACE 1300 through the Chromatography Data System, see Chapter 4, "Configuration and Method Parameters Setup from CDS."

# **TRACE 1300 User Interface**

The TRACE 1300 is not provided with an independent keyboard and display. It is fully controlled by the computer through a Thermo Scientific Chromatography Data System (CDS), which is also used for data handling and interpretation of the acquired results.

The user interface of the TRACE 1300 gas chromatograph is the **status panel** shown in Figure 32. It contains three **light emitting diodes** (LED), showing the current instrument's status, and the **Start/Stop** and **Maintenance** function keys.





See Chapter 3, "TRACE 1300 User Interface." and Chapter 4, "Configuration and Method Parameters Setup from CDS." for details.

# **TRACE 1310 User Interface**

This chapter describes the user interface of your TRACE 1310.

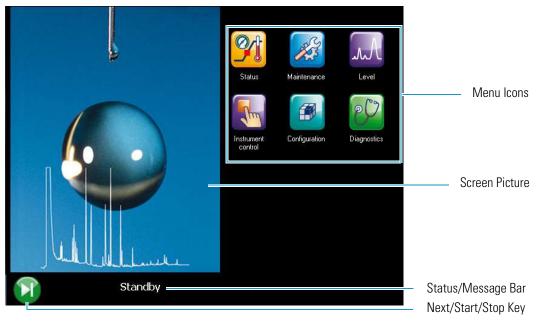
#### Contents

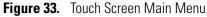
- TRACE 1310 User Interface Overview
- Configuring the Instrument
- Creating a Method
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- Performing a Maintenance

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# **TRACE 1310 User Interface Overview**

The TRACE 1310 user interface is a **touch screen** that provides immediate and detailed status display and guides you for ease of use and local control. When the TRACE 1310 is powered on, the touch screen main menu appears. See Figure 33.





#### **Menu Icons**

Each instrument's function is associated with an icon that opens the relevant menu as detailed below:

- Select the **Configuration** icon to configure your system. See "Configuring the Instrument" on page 33.
- Select the **Instrument Control** icon to program and visualize the method parameters for Oven, Front Inlet, Back Inlets, Front Detector, Back Detectors, Auxiliary Zones, and valves. See "Creating a Method" on page 51.
- Select the **Status** icon to monitor the status of the instrument. See "Monitoring the Instrument Status" on page 59.
- Select the **Level** icon to visualize the signal level of the front and back detectors. See "Monitoring the Signal Level" on page 63.
- Select the **Diagnostic** icon to bring up diagnostic information. See "Performing a Diagnostic" on page 64.
- Select the **Maintenance** icon when the maintenance of one or more components of your GC is required. See "Performing a Maintenance" on page 68.

### **Active/Deactive Icons**

Each menu visualizes a series of icons corresponding to the relevant module or component.

• When a module or component is installed on the GC system, the corresponding icon is colored and available for opening the relevant menu.



Example of **active** icon:

• When a module or component is not installed on the GC system, the corresponding icon is printed gray indicating that the relevant menu is unaccessible.

Example of **deactive** icon:

### **Data Entry Keys**

A **keyboard** appears on the right pane of the **Instrument Control** and **Status** menu when you press the name of a parameter or either an actual or setpoint field.

The numeric keyboard includes numbers from 0–9. The numeric keyboard includes a decimal point, and a minus key. The minus key acts as a negative sign (for entering sub-ambient temperatures), and as a range key (for entering sets of numbers). See Figure 34.





- Use the **Enter** key to confirm the entry or the modification done.
- Use the **Clear** key to erase the setpoint value or the modification done.
- Use the **Back** key to erase the last digit typed.
- Use the **Off/On** key to set the setpoint value to Off or On.
- Use the **>** key to close the keyboard.

#### ✤ How to entry or modify a parameter

To entry or modify a parameter, proceed as follows:

- 1. Press the name of parameter to enter or modify it, for example **Temperature**. The value in the setpoint field highlights.
- 2. Type the desired value, for example **250**, and then press **Enter** to confirm the modification.

**Note** If the **Actual** or **Setpoint** field has been selected, before typing the desired value, erase the highlighted value by pressing **Clear**.

#### **Shortcut Keys**

The shortcut keys are visualized on the status/message bar of each menu and submenu.

The short keys are:



**Go Back** — to return to the previous menu.

Go to Status — to jump immediately to the Status menu.

Go to Level — to jump immediately to the Level menu.

#### **Cursor Keys**

These keys indicating when not-currently visible menu items exist. They appear in three ways:

- Down arrow key \_\_\_\_\_ indicates that you can scroll downward.
- **Up arrow** key indicates that you can scroll upward.
- **Phase** key indicates that you can see the additional lines of the **Phase** menu when a PTV or a PTVBKF injector is used.

#### Hourglass



The hourglass indicates you should wait because the system is performing an action or a setting.

# **Configuring the Instrument**

The TRACE 1310 has been configured to your specifications at the factory. You might need to reconfigure the instrument when you make changes to the components. You must reconfigure the system when adding new components or changing injector modules, detector modules, carrier gases, and column types.

Press the **Configuration** icon on the touch screen main menu to configure the TRACE 1310. See Figure 35.



Figure 35. Touch Screen Main Menu: Configuration

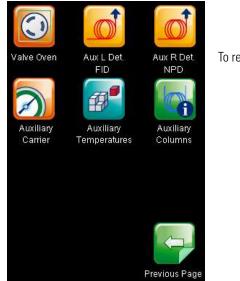
The Configuration menu appears. See Figure 36.



Figure 36. Configuration Menu - First Page

The icon More... appears to access the second page of the Configuration menu when additional modules are installed. See Figure 37.

Figure 37. Configuration Menu - Second Page





To return the previous page press the icon Previous Page

Press the desired icon to open the relevant configuration menu. See the following sections:

- "Oven Menu" on page 35
- "Back and Front Inlet Menu" on page 36
  - Configuring a SSL Injector
  - Configuring a SSLBKF Injector
  - Configuring a PTV Injector
  - Configuring a PTVBKF Injector
  - Configuring a GSV Injector
  - Configuring a HeS-SSL Injector
- "Back, Front, Aux L, and Aux R Detector Menu" on page 38
  - Configuring a FID Detector
  - Configuring a NPD Detector
  - Configuring a TCD Detector
  - Configuring an ECD Detector
  - Configuring a FPD Detector
  - Configuring a PDD Detector

- Configuring a GDI Detector
- "Analog Output Interface Menu" on page 41
- "Back and Front Column Menu" on page 41
- "Performing Column Evaluation" on page 43
- "Handshaking Menu" on page 44
- "Time/Units Menu" on page 45
- "Touch Screen Menu" on page 45
- "Network Menu" on page 46
- "Valve Oven Menu" on page 47
- "Aux Left and Right Detectors Menu" on page 48
- "Auxiliary Carrier Menu" on page 48
- "Auxiliary Temperature Menu" on page 49
- "Auxiliary Column Menu" on page 49

### **Oven Menu**



**Oven** — Sets preparatory actions of the oven.

- Auto prep run The choices are **On/Off**. Set **On** to turn on automatic Prep Run execution without pressing Prep Run.
- Auto Start It turns on the automatic start execution without pressing the start key. The choices are **On/Off**.
- **Prep run timeout** —Set the duration of the prep run. Enter a value in the range of 0–999.99 min. The injection must occur within this time otherwise this parameter will return the GC to the Standby condition.
- **Cryogenic Type** Enable or disable the cryogenic system when it is installed and configured with Carbon Dioxide or Liquid Nitrogen as a coolant. Select between N<sub>2</sub>, CO<sub>2</sub>, none.
  - Cryo timeout Enter the time at which the cryo system will be disabled.
     Enter a value in the range of 0–999.99 min.
  - Cryo Threshold Specify the temperature at which the cryo system begins to supply the coolant. Enter a value in the range of 40-200 °C.
- **Equilibration time** Set the time required for equilibrating the oven temperature after the temperature is set or modified. Enter a value in the range is 0–999.99 min.

- **Ready delay** Set the delay time before the GC enters the Ready to Inject condition. Enter a value in the range of 0–99.9 min. This time must not exceed the **Prep run timeout** value.
- **Oven max temp** Set the maximum allowable oven temperature to protect the column from unintentionally high temperatures. Enter a value in the range of 0–450 °C according to the manufacturer's maximum recommended operating temperature for the column.

# **Back and Front Inlet Menu**



Front Inlet Back/Front Inlet — Configures the injector module installed into the back/front position.

The TRACE 1310 works with the following types of injectors:

- SSL (Split/Splitless injector)
- SSLBKF (Split/Splitless Injector for backflush application)
- PTV (Programmable Temperature Vaporizing injector)
- PTVBKF (Programmable Temperature Vaporizing injector for backflush application)
- GSV (Gas Sampling Valve)
- HeS-SSL (Helium Saver)

### **Configuring a SSL Injector**

Set the following parameters:

- **Purge Flow** Enter a value in the range of 0.5-50 mL/min.
- **Gas Type** Select the carrier gas in use for the column. The choices are He, H<sub>2</sub>, N<sub>2</sub>, Ar/CH<sub>4</sub>, Ar, none.

### **Configuring a SSLBKF Injector**

Set the following parameters:

- **Purge Flow** Enter a value in the range 0.5-50 mL/min.
- Gas Type Select the carrier gas in use for the column. The choices are He,  $H_2$ ,  $N_2$ ,  $Ar/CH_4$ , Ar, none.
- **Backflush yes/no** To enable/disable the backflush.

### **Configuring a PTV Injector**

Set the following parameters:

- **Purge Flow** Enter a value in the range of 0.5-50 mL/min.
- Gas Type Select the carrier gas in use for the column. The choices are He,  $H_2$ ,  $N_2$ ,  $Ar/CH_4$ , Ar, none.

The following options determine the parameters to activate in the Injection Phase menu:

- **Cryogenic Type** Enable or disable the cryogenic system when it is installed and configured with CO<sub>2</sub> or LN<sub>2</sub> as a coolant. Select between N2, CO2, or none.
  - Cryo timeout Enter the time at which the cryo system will be disabled. Enter a value in the range of 0–30 min.
  - Cryo Threshold Specify the temperature at which the cryo system begins to supply the coolant. Enter a value in the range of 40-200 °C.
  - Cryo Cool at Select when cryo-cooling the instrument. Choose between Prep-Run or Post -Run.
- Evaporation Phase? yes/no Activates/deactivates the Evaporation Phase parameters.
- Cleaning Phase? yes/no Activates all the Cleaning Phase parameters. This control is not available for any CT mode.
- Ramped Pressure? yes/no Activates/deactivates all the Pressure parameters listed in the Injection Phases when the injection mode is set to Splitless. The range is On; Off.

#### **Configuring a PTVBKF Injector**

Set the following parameters:

- **Purge Flow** Enter a value in the range of 0.5-50 mL/min.
- Gas Type Select the carrier gas in use for the column. The choices are He,  $H_2$ ,  $N_2$ ,  $Ar/CH_4$ , Ar, none.

Use the following options to determine the parameters to activate in the Injection Phase menu:

- **Cryogenic Type** Enable or disable the cryogenic system when it is installed and configured with CO<sub>2</sub> or LN<sub>2</sub> as a coolant. Select between N<sub>2</sub>, CO<sub>2</sub>, or none.
  - Cryo timeout Enter the time at which the cryo system will be disabled. Enter a value in the range to 0–30 min.
  - Cryo Threshold Specify the temperature at which the cryo system begins to supply the coolant. Enter a value in the range 40-200 °C.
  - Cryo Cool at Select when cryo -cooling the instrument. Choose between Prep-Run or Post -Run.
- Evaporation Phase? yes/no Activates/deactivates the Evaporation Phase parameters.

- Cleaning Phase? yes/no Activates all the Cleaning Phase parameters. This control is not available for any CT mode.
- **Ramped Pressure**? **yes/no** Activates/deactivates all the Pressure parameters listed in the **Injection Phases** when the injection mode is set to **Splitless**. Select On or Off.
- **Backflush while Clean? yes/no** Enables/disables the backflush during the cleaning phase.
- Backflush on Inj./Evap Enables the backflush on injection and evaporation phases.
- **Backflush in CT** Enables the backflush in CT modes.

### **Configuring a GSV Injector**

Set the following parameters:

- Gas Type Select the carrier gas in use for the column. The choices are He,  $H_2$ ,  $N_2$ ,  $Ar/CH_4$ , Ar, none.
- Backflush enable yes/no Enables/disables the backflush.

#### **Configuring a HeS-SSL Injector**

Set the following parameters:

- **Purge Flow** Enter a value in the range of 0.5-50 mL/min.
- Gas Type Select the carrier gas in use for the column. The choices are He,  $H_2$ ,  $N_2$ ,  $Ar/CH_4$ , Ar, none.
- Enable Conservation Enables/disables the helium conservation feature.
- **Helium Delay** Set the delay time before the helium conservation feature starts up. Enter a value in the range 0.00-999.99 min.

# Back, Front, Aux L, and Aux R Detector Menu



**Back/Det Back/Front/Aux L/Aux R Detector** — Configures the detector module installed into the back/front position of the GC, and into the left/right auxiliary position of the TRACE 1310 Auxiliary Oven when present.

The TRACE 1300/TRACE 1310 works with the following types of detectors:

- FID (Flame Ionization Detector)
- NPD (Nitrogen Phosphorus Detector)
- TCD (Thermal Conductivity Detector)
- ECD (Electron Capture Detector)

- FPD (Flame Photometric Detector)
- PDD (Pulsed Discharge Detector)
- GDI (Generic Detector Interface)

To change a detector, mount and connect the detector module, then configure the GC and the data system, and plumb the appropriate gas supplies.

#### **Configuring a FID Detector**

Choose the type of **make-up** gas between **He** and **N**<sub>2</sub>.

#### **Configuring a NPD Detector**

Set the **polarization voltage** from 1 to 100 V.

Choose the type of **make-up** gas between **He** and **N**<sub>2</sub>.

#### **Configuring a TCD Detector**

Choose the carrier source between Front and Back.

#### **Configuring an ECD Detector**

Choose the type of **make-up** gas between N<sub>2</sub> and Ar/5%CH<sub>4</sub>.

Choose a **reference current** from 0.1 to 1.5 nA.

Choose a **pulse amplitude** from 5 to 50 V.

Choose a **pulse width** from 0.1 to 2.0  $\mu$ s.

#### **Configuring a FPD Detector**

Set the PMT voltage -800 or -1200 V.

#### **Configuring a PDD Detector**

PDD detector does not require configuration.

#### **Configuring a GDI Detector**

Set the parameters a third-party detector installed into the Generic Detector Interface.

**WARNING** The GDI electrical interface factory set-up includes three flow restrictors providing a full scale of 50 mL/min of Nitrogen. All three channels are set to **Nitrogen**, and 50 mL/min **full scale** by default.

If a different gas is used, it must be configured, and the full scale also changed accordingly. Use the following gas conversion factors to calculate the new full scale.

- He = 1.10
- H<sub>2</sub> = 2.20
- Air = 0.96
- Ar = 0.84
- $Ar/CH_4 = 0.86$

- To obtain the new full scale, multiply the  $\rm N_2$  full scale (50 mL/min) by the conversion factor:
- He = 50.0 \* 1.10 = 55.0
- $H_2 = 50.0 * 2.20 = 110.0$
- Air = 50.0 \* 0.96 = 48.0
- Ar = 50.0 \* 0.84 = 42.0
- $Ar/CH_4 = 50.0 * 0.86 = 43.0$

If higher flow rates or very low flow rates are required, the flow restrictor must be replaced by custom one. When a custom flow restrictor is used, the new full scale must be determined. The module is designed to use 1/4-in. encapsulated porous metal flow restrictors.

For replacing the encapsulated flow restrictor and determining the new full scale, refer to the sections **Adding a Generic Detector Interface | Replacing the Flow Restrictors** in Chapter 9 of the *TRACE 1300/TRACE 1310 Hardware Manual*.

- Gas#1 Select the type of gas in use connected to the Gas 1 line. The choices are Air,  $H_2$ ,  $N_2$ , He, Ar, and Ar/CH<sub>4</sub>. The default gas is  $N_2$ . Set the Full Scale value in the range from 1 to 1000 mL/min according to the Gas 1 flow restrictor. The default Full Scale is 50 mL/min.
- Gas#2 Select the type of gas in use connected to the Gas 2 line. The choices are Air,  $H_2$ ,  $N_2$ , He, Ar, and Ar/CH<sub>4</sub>. The default gas is  $N_2$ . Set the Full Scale value in the range from 1 to 1000 mL/min according to the Gas 1 flow restrictor. The default Full Scale is 50 mL/min.
- **Gas#3** Select the type of gas in use connected to the **Gas 3** line. The choices are Air,  $H_2$ ,  $N_2$ , He, Ar, and Ar/CH<sub>4</sub>. The default gas is  $N_2$ . Set the **Full Scale** value in the range from 1 to 1000 mL/min according to the Gas 1 flow restrictor. The default **Full Scale** is 50 mL/min.
- **ADC Scale** Set the Analog Digital Converter input full scale. The choose are 1 V, 5 V, and 10 V.

• **Temperature** — Set the detector temperature in the range from room temperature to 450 °C.

# **Analog Output Interface Menu**



Analog Output — Sets the configuration parameters of the Analog Output Interface. Up to four channels can be selected. For each channel of interest define the position of the detector, its attenuation range, and the Out Start Run signal. See also Using AOI Parameters in Chapter 19.

Trace 1310-Beta 1311							×
_	GC Conf	igura	tion	(A	nalog Outp	out)	
Channel #1	Det select	ion •	Ra 0	nge T			
Channel #2	Back	٠	1	-	Aux Oven	Aux L Det	Aux R Del
Channel #3	Right	•	2	•	Accilian	Auxiliary Temp.	Auxiliary
Channel #4	Left	٠	3	٠	Carrier		Columns
Out Start Run	<mark>High t</mark> a	o Lov	N	•	Analog Output		
$\mathbf{\widehat{N}}$	Standb	by .			50 °	- M 🕉	Previous

- **Det. selection** Select the position of the detector connected to the channel. Choose one: **Front** or **Back** if the detector is installed on the GC. Choose one **Left** or **Right** if the detector in installed on TRACE 1310 Auxiliary Oven.
- **Range** Select the range of the signal attenuation. Choose one: **0**, **1**, **2**, or **3** (10<sup>0</sup>, 10<sup>1</sup>,  $10^2$ ,  $10^3$ ).

**Note** The attenuation signal can be modified at a given time in the **Run Table** of the GC's user interface. See Run Table on page 54.

• Out Start Run — Select how the Out Start Run signal will change. Choose High to Low or Low to High.

# **Back and Front Column Menu**



Back Column Front Column Back/Front Column — Evaluates the column in use on the back/front position. Refer also to "Performing Column Evaluation" on page 43.



Set the following parameters:

- **Description** Enter a description of the column if desired.
- **Column length (m)** Enter the length of the column.
- Column nominal ID (mm) Enter the internal diameter of the column.
- Film thickness (µm) Enter the film thickness of the column.
- **Pre Column? (yes/no)** If a precolumn is present select **yes**, otherwise select **no**. If a precolumn is present, the menu requires users to set the length and the nominal internal diameter of the precolumn in the same ranges valid for the column.
- Pre Column Length (m) Enter the length of the precolumn.
- Pre Column ID (mm) Enter the internal diameter of the precolumn.
- **Post Column (yes/no)** If a post column is present select yes, otherwise select **no**. If a post column is present, the menu requires user to set the length and the nominal internal diameter of the post-column in the same ranges valid for the column.
- Post column length (m) Enter the length of the post-column.
- Post Column ID (mm) Enter the internal diameter of the post column.
- Calculated ID It indicates the calculated internal diameter of the column. See also K factor and Evaluation icon.
- **Column K Factor** The system calculates and shows the theoretical column K-factor according to the nominal dimensions of the column.

It is used to operate in flow mode. The deviation of the actual column ID from the nominal value is mostly affecting the column flow rate. To assure the utmost accuracy for the column carrier flow rate calculation, the Column Evaluation procedure is recommended.

# **Performing Column Evaluation**

This function allows the calculation of the actual column ID according to the actual flow of the carrier gas measured at the outlet of the column. The procedure requires the use of the column-flowmeter connector and of the digital flowmeter. Refer to the *TRACE 1300 and TRACE 1310 Hardware Manual* for details.

• **Column Evaluation** — Opens the column evaluation page.

 GC Configuration (Back Column)

 Column evaluation
 Image: Comparison (Comparison (Co

Press **Start** to begin the column evaluation routine.

Note To abort column evaluation press the Cancel button.

When the column evaluation is in progress, the system asks for the measured column flow rate. Enter this value in the range 0.5-10 mL/min, then press the **Apply** button to confirm.

In case of successful operation, the message **completed successfully** is shown.

The calculated ID is displayed, which reflects the actual column pneumatic resistance, permitting a more accurate calculation of the carrier gas linear velocity.



• **Save** — This command saves the nominal dimensions of your column into the USB stick. In the visualized page, select a line from 1 to 9 where you will save the data, then press **Ok**.

ol. In. 23	Nom. ID 0.400 - - -	Calc. ID 0.000 - - -	Film thk. 0.50 - -
23			
00	0.000	0.000	0.00
2.10	0.250	0.000	0.25
n: -	Ok		Cancel
	.10	00 0.000 1.10 0.250 - Ok	00 0.000 0.000 .10 0.250 0.000 - Ok

• Load — Loads the nominal dimensions of a column previously saved on the USB stick. In the visualized page, select the line where the data to load are saved, then press Ok.

# **Handshaking Menu**



Hand Shaking — Configures the signals from external devices, such as an autosampler or a mass spectrometer, during the analysis. To allow other devices to run properly, you must indicate how the signal will change.

- **Remote Start** Allows another device to start the GC. The choices are **L to H** (low to high) or **H to L** (high to low). For the AI 1310/AS 1310 autosampler, you must select **H to L**.
- Inhibit Ready Delays readiness until the GC receives a signal from another device. The choices are L (low), H (high), or Neither.
- End of Run Signals another device that the run has ended. The choices are L to H (low to high), or H to L (high to low).
- **Start of Run** Signals another device, that the run has started. The choices are **L to H** (low to high), or **H to L** (high to low).
- **Ready Out** Signals another device that the GC is ready. The choices are L (low), or H (high).
- **Prep Run** Signals another device that the GC is preparing for a run. The choices are L (low), or H (high).

# **Time/Units Menu**



Time/Units **Time/Units** — Sets the time, the date, and allows choosing the pressure unit.

- **Date** Enter the date (mm.dd.yyyy) by using the numeric keyboard.
- **Time** Enter the time (hh.mm) on a 24-hour clock by using the numeric keyboard. For example, for **8:05 a.m.**, enter **08.05**; for **2:30 p.m.**, enter **14.30**.
- **Pressure Unit** Choose the pressure unit among **psi**, **kPa** and **bar**.

### **Touch Screen Menu**



Touch screen Touch Screen — Allows you to choose a language, adjust the brightness and volume, customize the panel back-color, replace the screen picture, and calibrate the display. See Figure 38.

Figure 38. Touch Screen Menu



- **Language** English is the default language. Choose a language among those available. Once a language is selected, the menus and all other windows, functions, and messages will be in that language.
- **Brightness** Adjusts the brightness of the screen. Use the left/right arrows respectively to decrease/increase the brightness level.
- **Volume** Adjust the volume of the sound heard when a icon is pressed. Use the left/right arrows respectively to decrease/increase the volume level.

- Main menu screen saver Causes blanking the screen after a period of inactivity. Select On or Off. When On the function is enabled; when Off the function is disabled.
- **Panel Backcolor** The grey is the default backcolor. Choose a backcolor among those available in the color palette.





**Restore Default Screen** — Replaces the default screen picture. Loads a new screen picture, in \*.**bmp** format (346 pixels long x 386 pixels high), from a USB stick plugged into the USB port under the touch screen box.



**Display Calibration** — Press this icon to calibrate the screen in function of the tool used to press it. A cross appears in the center of the visualized white page. Press the cross with a pen or with a finger of your hand; continue to press the cross according to the instruction displayed on the top of the page. The cross disappears when the calibration is complete. At the end of the calibration, exit the page by pressing the screen in any point.

# Network Menu



**Network** — Configures the Network parameters to allow LAN control of the GC through a Thermo Scientific chromatography data systems. See Figure 39.

#### Figure 39. Network Menu GC Configuration (Network) IP Address 10.209.90.94 7 8 9 5 255.255.0.0 4 0.0.0.0 5 6 DHCP Enabled No 2 3 1 2551 0 Enter ? 79 °C M 🦅 N Standby

The TRACE 1300/TRACE 1310 is shipped with a default IP address that might not match the needs of the LAN where the GC must be installed. To change the default values, contact your LAN administrator and ask the IP address to be assigned, the netmask, and eventually the port.

- The IP address is a 3 digits x 4 fields given by the network administrator, e.g. 192.168.127.10.
- The netmask is a 3 digits x 4 fields given by the network administrator e.g. 255.255.255.0
- The port is a number given by the network administrator, e.g. 2551.

When the parameters required are set, press Apply.

# Valve Oven Menu



Valve Oven Valve Oven — Configures the heating zones of the TRACE 1310 Auxiliary Oven. See the example of Figure 40.

Figure 40. Valve Oven Menu



- Heater 1: Select the component connected to the heater 1 among Valve Oven, H. T. Valve Oven, and Aux 3.
- Heater 2: Select the component connected to the heater 2 among Aux Col. Oven, Aux 4, and Methanizer.

**Note** The configuration of the heater type determines label in UI and temperature ranges.

# **Aux Left and Right Detectors Menu**



FID Aux L Det/Aux R Det — Configures the auxiliary detector module installed into the left/right position on the TRACE 1300 Auxiliary Oven (Valve Oven). The configuration menu is the same of the detector modules installed in front and back positions on the GC. See "Back, Front, Aux L, and Aux R Detector Menu" on page 38 for details.

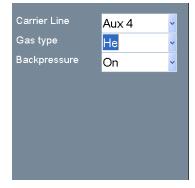
# **Auxiliary Carrier Menu**



**Auxiliary Carrier** — Sets the parameters for the auxiliary gas modules.

This menu includes both the Auxiliary Gas modules. One of them will be mounted inside the Valve Oven module, and the other into the GC. See Figure 41.





- **Carrier line** Select the auxiliary carrier line **Aux 1-3**, **Aux 4-6**, or both Auxiliary Gas modules to give **Aux 1-6**.
- **Gas type** Select the type of the carrier gas in use for the selected auxiliary carrier line. The choices are He, H<sub>2</sub>, and N<sub>2</sub>.
- **Back pressure** Select the back pressure function. The choices are Yes, No. Select **Yes** if the carrier line is used as vent.

# **Auxiliary Temperature Menu**



Auxiliary Temperature — Configures the heater zones of the Auxiliary Temperature/Cryo module. See Figure 42.

Figure 42. Auxiliary Temperature Menu



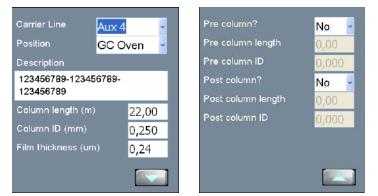
- Heater 1: Select the component connected to the heater 1 between X-Line 1, Aux 1, and None.
- Heater 2: Select the component connected to the heater 2 between X-Line 2, Aux 2, and None.

# **Auxiliary Column Menu**



**Auxiliary Column** — Details the auxiliary column in use. See Figure 43.

Figure 43. Auxiliary Column Menu



The Aux **1-6** auxiliary carrier column settings not have the field for measuring the column K Factor. The theoretical value calculated from the column parameters is only use.

Set the following parameters:

- **Carrier Line** Select the auxiliary carrier line at which the auxiliary column is connected.
- **Position** Indicate where the auxiliary column is added. Choose among **GC Oven**, **Valve Oven**, **Aux C. Oven**.
- **Description** Enter a description of the column if desired.
- **Column length (m)** Enter the length of the column.
- Column ID (mm) Enter the internal diameter of the column.
- Film thickness (µm) Enter the film thickness of the column.
- **Pre Column? (yes/no)** If a precolumn is present select **yes**, otherwise select **no**. If a precolumn is present, the menu requires users to set the length and the nominal internal diameter of the precolumn in the same ranges valid for the column.
- Pre Column Length (m) Enter the length of the precolumn.
- Pre Column ID (mm) Enter the internal diameter of the precolumn.
- **Post Column (yes/no)** If a post column is present select yes, otherwise select **no**. If a post column is present, the menu requires user to set the length and the nominal internal diameter of the post-column in the same ranges valid for the column.
- Post column length (m) Enter the length of the post-column.
- Post Column ID (mm) Enter the internal diameter of the post column.

# **Creating a Method**

Here you can program the control parameters for all the modules present in your system.

**Note** For information about setting control parameters for other instruments and software, refer to the appropriate manuals.

Press the **Instrument Control** icon on the touch screen main menu for programming the parameters in the analytical method. See Figure 44.



Figure 44. Touch Screen Main Menu: Instrument Control

The Instrument Control menu appears. See Figure 45.



Figure 45. Example Instrument Control Menu

The **Instrument Control** menu lets you program and visualize the parameters for the oven, inlets, detectors, auxiliary modules, and valves when installed. Through the **Run Table** page you setup detectors, valves, and external events to occur at the Prep-run or during a run.

Press the icon to open the relevant submenu where the setpoint parameters can be set through the numerical keyboard that appears on the right panel when you press the name of the parameters, or either the actual and setpoint fields.



**CAUTION** The parameters cannot be programmed when the analysis is running.

Press the desired icon, to open the relevant submenu. See the following sections:

- "Oven Parameters" on page 52
- "Back and Front Inlet Parameters" on page 52
- "Back and Front Detector Parameters" on page 53
- "Aux Left and Right Detectors Parameters" on page 53
- "Run Table" on page 54
- "Auxiliary Temperatures" on page 55
- "Valves" on page 56
- "Valve Oven" on page 56
- "Auxiliary Carrier" on page 57

### **Oven Parameters**



**Oven Oven** — This section lists the parameters for programming the oven temperature, from an initial temperature to a final temperature, using up to 32 ramps during the analytical run. It is possible to set a single (isothermal) or multiple ramp programs. For a detailed description see the section Using Oven Parameters in Chapter 5.

# **Back and Front Inlet Parameters**



Front Inlet Back/Front Inlet — This section lists the parameters for programming the injector modules installed into the back/front position:

- Split/Splitless Injector (SSL). See Using SSL Parameters in Chapter 6.
- Split/Splitless Injector for Backflush applications (SSLBKF) See Using SSLBKF Parameters in Chapter 7.

- Gas Sampling Valve (GSV) See Using GSV Parameters in Chapter 8.
- Helium Saver (HeS S/SL) See Using Instant Connect Helium Saver Injector Module Parameters in Chapter 9.
- Programmable Temperature Vaporizing Injector (PTV) See Using PTV Parameters in Chapter 10.
- Programmable Temperature Vaporizing Injector for Backflush application (PTVBKF). See Using PTVBKF Parameters in Chapter 11.

# **Back and Front Detector Parameters**



**BackDet** Front Det Back/Front Detector— This section lists the parameters for programming the detector modules installed into the back/front position:

- Flame Ionization Detector (FID) See Using FID Parameters in Chapter 12.
- Nitrogen Phosphorus Detector (NPD) See Using NPD Parameters in Chapter 13.
- Thermal Conductivity Detector (TCD) See Using TCD Parameters in Chapter 14.
- Electron Capture Detector (ECD) See Using ECD Parameters in Chapter 15.
- Flame Photometric Detector (FPD) See Using FPD Parameters in Chapter 16.
- Pulsed Discharge Detector (PDD) See Using PDD Parameters in Chapter 17.
- Generic Detector Interface (GDI) See Using GDI Parameters in Chapter 18.

# **Aux Left and Right Detectors Parameters**



**FID Aux L Det/Aux R Det** — This section lists the parameters for programming the detector modules installed into the left/right position on the TRACE Auxiliary Oven (Valve Oven). The parameters menu is the same of the detector modules installed in front and back position on the GC. See "Back and Front Detector Parameters" on page 53 for details.

# **Run Table**



Run Table — Sets detectors, valves, and external events to occur at the Prep-run or during a run. See the example in Figure 46.

Figure 46. Run Table Menu

Time	Item	Action		+	+
Prep-run	Aux Gas #3	55.0			
Prep-run	FID Front	Auto Zero		Back Inlet	
7.50	Aux Gas #1	520.0	Oven	PTV	Front Intel SSL
8.00	Aux Gas #2	90.0		<b></b>	+
10.00	Aux Gas #1	60.0	100		
10.00	Aux Gas #2	5.0		Back Det	
11.00	FID Front	Auto Zero	Run Table	FID	
			<b>#</b>	D	O
			Auxiliary Temperatures	Aux L Det. FID	Aux R Del
Add	Modify	Remove		0	
	-		Valves	Valve Oven	Aux. Carrie
	Not	Ready	44 °C	MA 9	ተ 🏠

Events displayed on the Run Table page are from selections made in the **Run-Time Event** screen. See Figure 47.

The timetable has three columns: Time, Item, and Setting. Events are sorted into time order automatically.

**Time** — Shows the time in the run at which the event will take place.

Item — Shows the event type and identifies the external event, or valve number.

Action — Indicates the action associated with the event.

Events can be added, modified, or removed by using the corresponding **Add**, **Modify**, and **Remove** buttons.

The Add button opens the **Run-Time Event** screen. See the example in Figure 47.

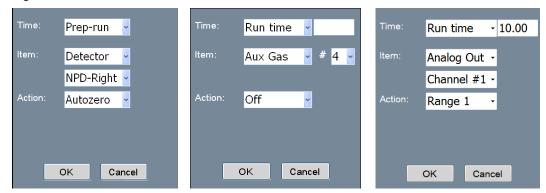


Figure 47. Run-Time Event Screen

**Time** — Select either the **Prep Run** or **Run Time** option. When you select the Prep Run option, the time field is disabled. Select the Run Time option to enable the adjacent field. Enter the time at which the event is to occur in the range is 0.00-999.99 min,

Item — Detector, Aux Gas, Valve, Valve Oven, and Analog Out are the types of event. Select the required event to activate the associated fields.

Action — Enables the action appropriate to the type of event.

The event options are:

- Autozero Applied to all detector types.
- Neg. Polarity Applied to TCD only. The entry will invert the polarity at the given time.
- **Source Current** Applied to NPD only. The entry will change the source current at the given time.
- **On** Sets the event on.
- **Off** Sets the event off.
- Pressure Set the pressure event.
- **Range** Applied to the module Analog Output Interface only. The entry will change the range of the attenuation signal from  $10^0$  to  $10^3$  of the selected **Channel** at the given time.

# **Auxiliary Temperatures**



**Temperatures** Auxiliary Temperatures — Defines the temperature parameters for the external auxiliary temperature modules set in the configuration. See the example in Figure 48.

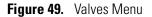


#### Figure 48. Auxiliary Temperature Menu

# Valves



Valves Valves — Sets On or Off for up to eight external valves connected to the Aux Temperature/Cryo Module. See the example in Figure 49.





A second Valve page is available to set **On** or **Off** up to eight valves installed into the Valve Oven (TRACE 1310 Auxiliary Oven) when present.

# **Valve Oven**



Value Oven Valve Oven — Sets the auxiliary temperatures of the TRACE 1310 Auxiliary Oven. See the example of Figure 50.



Figure 50. Valve Oven Menu

The temperature limits must be consistent with the configuration as for the auxiliary temperatures. See Table 3.

#### Table 3. Auxiliary Temperature Limits

Туре	Max Temperature °C	Available on:	Notes
Transfer Line	400	Aux 1, Aux 2	Primary module, display as Transfer Line.
Valve Oven	200	Aux 3 only	Secondary module (Valve Oven)
High Temperature Valve Oven	300	Aux 3 only	Secondary module (Valve Oven)
Aux Column Oven	250	Aux 4 only	Secondary module (Valve Oven)
Methanizer	400	Aux 4 only	Secondary module (Valve Oven)
Aux Temperature (generic)	400	All Aux 1, 2, 3, and 4	Primary and secondary modules, display as Aux Temperature (#)

# **Auxiliary Carrier**



Aux Carrier Aux Carrier — Sets up to six auxiliary carrier lines selected by the Carrier Line drop down menu. See the example of Figure 51.



#### Figure 51. Auxiliary Carrier Menu

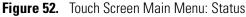
- **Carrier line** Select the auxiliary carrier line **Aux 1-3**, **Aux 4-6**, or both Auxiliary Gas modules to give **Aux 1-6**.
- Flow mode Defines the auxiliary carrier gas control mode to use: Constant Flow, Constant Pressure, Programmed Flow, and Programmed Pressure. Each mode activates or deactivates the dedicated parameters.

# **Monitoring the Instrument Status**

You can visualize the status of the run, the pressure status of the inlets, and the actual and setpoint status temperatures for oven, inlets, and detectors. However only the temperature setpoint can be modified.

Press the **Status** icon on the touch screen main menu to monitor the status of the TRACE 1310. See Figure 52.





The Status menu appears. See Figure 53.

Figure 53. Status Menu



Press the desired icon to open the relevant submenu. See the following sections:

- "Run Status" on page 60
- "Pressure Status" on page 61
- "Zone Temperature" on page 62
- "Waiting For" on page 62
- "Monitoring the Signal Level" on page 63

# **Run Status**



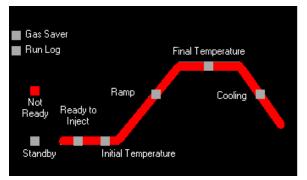
**Run Status** — Monitors the current time, data, oven temperature, last run time, next run, elapsed time, and how the TRACE 1310 status LEDs look during each stage in a run. See the example in Figure 54.

Figure 54. Run Status



The oven ramp LEDs indicate the temperature ramp stages during a run. You can follow the progress of a run by observing these LEDs and the relevant message in the Status bar. See Figure 55.





The stages in a run are:

- Not Ready The TRACE 1310 is waiting for one or more parameters to become ready.
- Standby All the GC parameters have reached the ready condition.

• **Preparing to run** — When the GC is in standby condition, start the prep run routine by

pressing the green start button on the lower left corner on the page. The GC sets all conditions for a run.

• **Ready to Inject** — The preparatory phase has finished indicating you can manually inject a sample, or start an autosampler. The green start button appears on right pane of the page. Press the green button to start the analytical run. See Figure 56.



Figure 56. Green Start Button

- Initial Temperature This LED lights up when a run starts and remain lit during the initial hold time.
- **Ramp** This LED lights up when the temperature begins rising in the first ramp and remains lit until the last ramp's temperature has been reached.
- **Final Temperature** This LED lights up when the last ramp's temperature is reached and stays on during the last ramp's holding time.
- **Cooling** The TRACE 1310 returns to initial temperature and pressure conditions during this stage.

Other LEDs are:

- Gas Saver This LED lights up when the gas saver function is enabled.
- **Run Log** This LED lights up when the Run Log contains entries. See "Run Log" on page 67.

# **Pressure Status**



Pressure status — Monitors the current pressure of each injector module installed. The visualized value is not modified.

# **Zone Temperature**



**Zone Temperature** — Monitors the actual and setpoint temperature of all TRACE 1300 heated zones. You can modify the temperature setpoint of the zone of interest through the numerical keyboard that appears on the right pane. See the example in Figure 57.





- The **green** background of the actual value indicates that it has reached the setpoint value programmed.
- The **red** background of the actual value indicates that the setpoint value has not yet been reached.

# **Waiting For**



**Waiting for** — This function monitors and displays the actions or conditions for which the GC is waiting for to go to ready status. See the list in the example in Figure 58.

Figure 58. Example of Waiting For Function

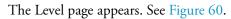


# **Monitoring the Signal Level**

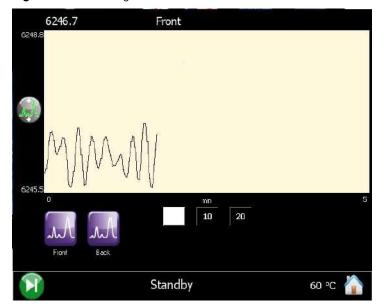
Press the **Level** icon on the touch screen main menu to monitor the signal level of the front and/or back detectors. See Figure 59.



Figure 59. Touch Screen Main Menu: Level



#### Figure 60. Level Page



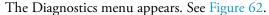
To see the signal level of the detector module of interest, press the **Front**, **Back**, **Aux L**, or **Aux R** icon.

# **Performing a Diagnostic**

Press the **Diagnostics** icon on the touch screen main menu for bringing up diagnostics information. See Figure 61.



Figure 61. Touch Screen Main Menu: Diagnostics



#### Figure 62. Diagnostic Menu



Press the desired icon to open the relevant submenu. See the following sections:

- "Software Info" on page 65
- "Hardware Info" on page 65
- "Network Info" on page 66
- "Readings" on page 66
- "Error" on page 66
- "Run Log" on page 67
- "Save to USB" on page 67

# **Software Info**



**Software Info** — Indicates the firmware version of GC, oven, and injector and detector modules. See the example in Figure 63.

Figure 63. Software Info Page

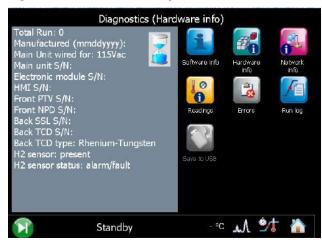


# **Hardware Info**

# **B**

**Hardware Info** — Indicates the number of total runs performed, the manufacturing date and the serial number of your GC, and the serial number of each component and module installed. See the example in Figure 64.

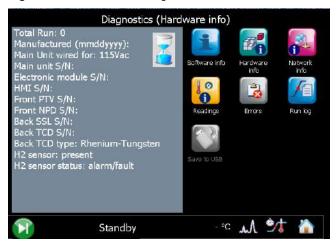
Figure 64. Hardware Info Page



# **Network Info**

Network Info — Indicates the network connection specifications (IP address, Subnet mask, etc), and suffix DNS description. See the example in Figure 65.

Figure 65. Network Info Page



# **Readings**

0 **Reading** — Displays the status of the various devices. See the example in Figure 66.

#### Figure 66. Readings Page

0



# Error



Data — Indicates the diagnostic of the possible errors.

# **Run Log**



**Run Log** — This function displays the run log, which records errors that happen during run time. It also displays the time and description of any deviations that occur.

The run log keeps track of any errors or deviations during the run. This information can be used to meet good laboratory practice (GLP) standards. For example, if you interrupt the run for any reason, the run log will record the time, the run stopped and an interpretation of the event.

When the run log contains entries, the Run Log Status LED is lit. To see the journal of events, press the **Run Log** icon. The Run Log is cleared and reset at the beginning of the next run.

# Save to USB

**Sav** 

**Save** — This command saves the all the contents of the Diagnostic sub-menus into the USB stick in text format. The filename is automatically created by the system and includes the name of the instrument, the date, and the time of the saving

# **Performing a Maintenance**

Press the **Maintenance** icon on the touch screen main menu to perform a required maintenance. See Figure 67.



Figure 67. Touch Screen Main Menu: Maintenance

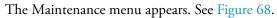
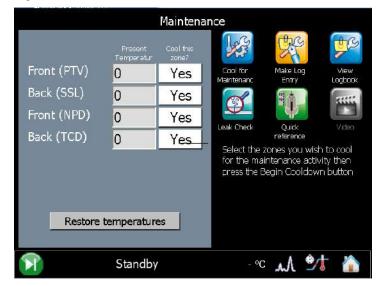


Figure 68. Maintenance Menu



Press the desired icon to open the relevant submenu. See the following sections:

- "Cool for Maintenance" on page 69
- "Make Log Entry" on page 69
- "View Logbook" on page 70

- "Leak Check" on page 70
- "Quick Reference" on page 71

# **Cool for Maintenance**



Cool For Maintenance — This function allows cooling of the system for maintenance. See Figure 69.



	Present Temperature	Cool this zone?			
Front (SSL)	79	No			
Back (PTV)	41	No			
Front (NPD)	8	No			
Back (FID)	26	No			
MS xfer line					
Ion Source		-			
Begin	Begin Cooldown				

In this page, select **Yes** for the zones you wish cool down for maintenance activity, then press **Begin Cooldown**.

# Make Log Entry



Make Log Entry — This function displays the maintenance log entry page. See Figure 70. Press the corresponding white buttons to mark all the maintenance performed. Press Enter into log to save the settings in the logbook. See also "View Logbook" on page 70.

Figure 70. Make Log Entry Page

Maintenance(Make Log Entry)					
Frant	Back				
V	V	Replaced septum	Cleaned ion source		
V	V	Replaced liner	Replaced filament		
		Trimmed column	Changed pump oil		
V	V	Cleaned inlet	Charged FC-43 vial		
		Replaced column	V Cleaned air filter		
		Replaced pre-colum			
V	V	Replaced carrier filter	Enter into log Exit		
F		Standby	-•c M 💁 🏠		

# **View Logbook**



**View Logbook** — This function displays a logbook which records the time and description of any maintenance performed. See Figure 71.

Figure 71. View Logbook Page

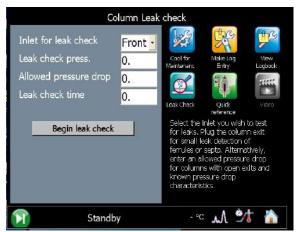
Maintenance(View Logbook)					
feb/28/2012 13:56 Replaced septum (Front)					
eb/28/2012 13:56 Replaced liner (Front)					
feb/28/2012 13:56 Cleaned inlet (Front)					
feb/28/2012 13:56 Replaced carrier filter (Front)					
feb/28/2012 13:56 Replaced septum (Back)					
feb/28/2012 13:56 Replaced liner (Back)					
feb/28/2012 13:56 Cleaned inlet (Back)					
feb/28/2012 13:56 Replaced carrier filter (Back)					
feb/28/2012 13:56 Cleaned air filter					
Delete Logbook Exit					
Standby - °C 🔊 🎌 🏠					

# Leak Check



**Leak Check** — This function allows to perform a leak check. See Figure 72. This procedure requires the use of the column-flowmeter connector. Refer to the *TRACE* 1300 and *TRACE* 1310 Hardware Manual for details.

Figure 72. Leak Check Page



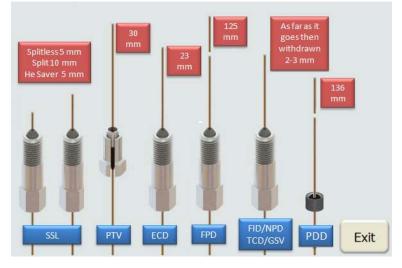
In this page, select the front/back inlet, set the pressure and the duration of the leak check, and then the allowed pressure drop. To start the routine, press **Begin leak check**.

#### **Quick Reference**



Quick Reference — This function opens the quick reference page. See Figure 73.

Figure 73. Quick Reference Page



This page shows the proper column insertion depths for SSL, PTV, ECD, FPD, FID, NPD, TCD, and GSV. Detector and injector insertion depths are measured from the top of the ferrule.

For PDD detector, the column must penetrate **136 mm** inside the capillary column adapter.

To exit this page, press Exit.

# **TRACE 1300 User Interface**

This section provides a description of the user interface of your TRACE 1300.

#### Contents

- TRACE 1300 User Interface Overview
- Configuring and Method Parameters Setup

# **TRACE 1300 User Interface Overview**

The user interface of the TRACE 1300 gas chromatograph is the **status panel** shown in Figure 74. It consists of three **light emitting diodes** (LED), showing the current instrument's status and the **Start/Stop** and the **Maintenance** function keys.

Figure 74. Status Panel



Every instrument's event/phase is associated with a status of the LED as detailed below:

#### 1. Power On the GC.

a. All the LED on the status panel light up simultaneously. Next the **Power** light becomes a solid green while all the other lights turn off. See Figure 75.

#### Figure 75. Power On



b. The GC goes to **stand-by** status. The **Power** light remains a solid green while all the other lights are off. See Figure 76.



#### 2. Analysis

a. Press the **Start/Stop** key. If the GC parameters have not yet reached the setpoint value, the **Ready** light blinks orange indicating the not ready status. See Figure 77.

Figure 77. Not Ready Status



When all the parameters are ready, the **Ready** light turns off, and the GC goes to **standby mode**. Only the **Power** light is on. See Figure 76.

b. Press the **Start/Stop** key. If all the parameters are ready, the GC enters the **PrepRun** (preparing for run) phase. The **Ready** light blinks green and fast. See Figure 78.

Figure 78. Preparing for Run



When the **Prep Run** phase is completed, the **Ready** light becomes a solid green. The GC goes to **Ready to Inject** mode. See Figure 79.

Figure 79. Ready to Inject



c. When the GC is in **Ready to Inject** mode, press the **Start/Stop** key. The **Ready** light will turn off while the **Run** light becomes a solid blue. The analytical run starts. See Figure 80.



**Note** To stop an analytical run in progress, press the **Start/Stop** key. This resets the GC from **Run** to **Not Ready** condition.

When the analytical run is completed, the **Run** light turns off and the GC enters in the cooling phase.

d. The **cooling** phase starts when the analytical run is completed or stopped, and restores the parameters to the initial setpoint. The **Ready** light blinks orange indicating the not ready status. See Figure 81.

Figure 81. Not Ready Status



When the cooling phase is completed, the **Ready** light turns off, and the GC goes to **standby** mode. Only the **Power** light is on. See Figure 76 on page 74. The GC is ready for a new analytical run.

#### 3. Maintenance

This function is not allowed during a run.

a. Press the **Maintenance** key and keep it pressed for three seconds. The light on the key blinks green indicating that the GC is cooling for maintenance. See Figure 82.

Figure 82. Maintenance (1)



All the heated zones cool at 60 °C. This function also performs the following actions automatically:

- turns off the flame and the fuel gases of the FID detector.
- turns off the thermionic source and the hydrogen and the air flows of the NPD detector.
- turns off the filaments of the TCD detector.
- b. When the GC is ready for maintenance, the light into the key turns solid green. See Figure 83.

Figure 83. Maintenance (2)



c. When maintenance is completed, press the **Maintenance** key. The light on the key turns off while the **Ready** light blinks orange indicating the not ready status. See Figure 84.

Figure 84. Not Ready Status

When all the parameters are restored, the **Ready** light turns off, and the GC goes to **standby** mode. Only the **Power** light is on. See Figure 76 on page 74.

#### 4. Error

a. When an alarm condition is detected, both **Power** and **Ready** lights are blinking, and a typical sound (beep) is heard. Power, temperatures, and gases are automatically cut off. See Figure 85.



#### 5. Loading Firmware

a. During the loader embedded firmware upgrade, the **Power** and **Maintenance** lights are solid green, the **Ready** light is off, and the **Run** light is solid blue. See Figure 86.

Figure 86. Loading Firmware



# **Configuring and Method Parameters Setup**

The TRACE 1300 is not provided with an independent keyboard and display. It is fully controlled by the computer through a Thermo Scientific Chromatography Data System (CDS) also used for data handling and interpretation of the acquired results.

To configure your GC system, tune the instrument, create methods, run samples, and explore your data, refer to the manuals of the relevant CDS in use.

For details, see Chapter 4, "Configuration and Method Parameters Setup from CDS,"

4

# **Configuration and Method Parameters Setup from CDS**

This chapter contains instructions to configure your TRACE 1300/TRACE 1310 and to edit the parameters (according to the front/back injector, front/back/aux L/aux R detector modules, and optional devices installed), through Chromeleon, Xcalibur, Chrom-Card, ChromQuest, or EzChrom, Thermo Scientific Chromatography Data System (CDS).

#### Contents

- Introduction
- Configuration General Tab
- Configuration Handshaking Signals
- Configuration Inlets Tab
- Configuration Detectors Tab
- Configuration Aux Oven (Valve Oven)Tab
- Configuration Auxiliary Temperature Tab
- Configuration Auxiliary Carrier Tab
- Edit Method Parameters
- Oven Page
- S/SL Page
- S/SL Backflush Page
- PTV Page
- PTV Backflush Page
- GSV Page
- Helium Saver (HeS-S/SL) Page
- FID Page
- ECD Page
- NPD Page
- TCD Page
- FPD Page
- PDD Page
- GDI Page
- Auxiliary Temperature Page
- Auxiliary Carrier Page
- Run Table Page
- Run-Time Event

**Note** Column Evaluation and Leak procedures are not included in this chapter. To perform them, please refer to the manual of the data system you are using.

# Introduction

Run your Chromatography Data System (Chromeleon, Xcalibur, Chrom-Card, ChromQuest, or EzChrom), then open the TRACE 1300 Configuration window. See the example in Figure 87.

TRACE 1300 Series GC Configuration		8 🗙
General Handshake Inlets Detectors Aux. C Name Device name: TRACE 1300 Connection Network address: 169 254 250 5 Advanced	Oven Aux. Temperature Aux. Carrier	OK Cancel
		Get

Figure 87. Configuration Window (Chromeleon CDS)

The configuration window is subdivided into the following tabs.

- "Configuration General Tab" on page 79
- "Configuration Handshaking Signals" on page 80
- "Configuration Inlets Tab" on page 80
- "Configuration Detectors Tab" on page 81
- "Configuration Aux Oven (Valve Oven)Tab" on page 83
- "Configuration Auxiliary Temperature Tab" on page 84
- "Configuration Auxiliary Carrier Tab" on page 85

The configuration window includes the following common buttons:

**Get** — Use this button any time you want to automatically enter the configurations already entered into your GC.

**Ok** — Use this button to close the dialog box and confirm your selection.

**Cancel** — Use this button to clears all modifications done.

Help — Use this button to open help instruction.

# **Configuration General Tab**

This tab allows you to configure the network address, the handshaking parameters, and the pressure unit.

This tab includes the following fields:

#### Name

This field is present only in Chromeleon CDS. Specifies the name of the device to configure.

#### Connection

In this field you set the LAN communication between the TRACE 1300 and the data system.

• Network Address — Enter the I.P. address to allow the LAN control of the GC through the Thermo Scientific CDS.

The TRACE 1300 is shipped with a default IP address that might not match the needs of the LAN where the GC must be installed.

To change the default values, contact your LAN administrator and ask for the IP address to be assigned, the netmask, and eventually the port.

- The IP address is a 3 digits x 4 fields number given by the network administrator e.g. 192.168.127.10
- Advanced Click on this button to open the Advanced LAN Setting window where the advanced parameters for LAN communication may be set.
  - **Port No** it specifies the port number.
  - Timeout (ms) it specifies the timeout value in milliseconds.
     Generally the two default values are 500 and 2551 respectively.

#### Option

This field includes the following options:

- Pressure Unit From the drop-down list, choose one: psi, kPa, or bar.
- Lock GC input during run Check this box if GC inputs are not desired during the run.

• Autostart — Select this check box if you want the GC start automatically when ready.

# **Configuration Handshaking Signals**

**Note** Chromeleon CDS, specifies the handshaking signal into a dedicated Handshake tab. Xcalibur, Chrom-Card, ChromQuest and EzChrom specifies the Handshake signal in the General tab.

Configures the signals from external devices, such as an autosampler or mass spectrometer, during analysis. To allow other devices to run properly, you must indicate how the signal will change.

- **Remote Start In** Allows another device to start the GC. The choices are **L to H** (low to high) or **H to L** (high to low). For the AI 1300/AS 1300, AI 3000/AS 3000, TriPlus, TriPlus RSH, or TriPlus 100 Liquid Sampler autosampler, you must select **H to L**.
- End of Run Out Signals another device that the run has ended. The choices are L to H (low to high) or H to L (high to low).
- Inhibit Ready Delays readiness until the GC receives a signal from another device. The choices are L (low), H (High) or Neither.
- GC Ready Out Signals another device that the GC is ready. The choices are L (low) or H (High).
- Start of Run Out Signals another device that the run has started. The choices are L to H (low to high) or H to L (high to low).
- **Prep Run Out** Signals another device that the GC is preparing for a run. The choices are L (low) or H (High).

# **Configuration Inlets Tab**

Use this tab to select the inlets installed on your GC and the carrier gas used.

#### Inlets

This field includes the following options:

- Inlet Type Choose the injector module installed on your GC.
  - Front Choose one: None, S/SL, S/SL with Backflush, Gas Sampling Valve, Helium Saver S/SL, PTV, and PTV Backflush.
  - Back Choose one: None, S/SL, S/SL with Backflush, Gas Sampling Valve, Helium Saver S/SL, PTV, and PTV Backflush.

- Carrier Gas Choose the type of carrier gas used to supply the front/back injector module.
- Front Choose one: Helium, Hydrogen, Nitrogen, Argon, or Argon/Methane.
- **Back** Choose one: Helium, Hydrogen, Nitrogen, Argon, or Argon/Methane.
- Name Type the front/back position the injector module is installed.

#### **Options**

Select Hydrogen sensor check box if a hydrogen sensor is installed into the GC.

## **Configuration Detectors Tab**

Use this tab to configure the detectors you will use with your TRACE 1300/1310.

If you change your detectors configuration, remember to reconfigure your TRACE 1300/1310 here. The detectors chosen here will give rise to related pages and will be listed in the Detector Events located in the Run Table Page.

This tab includes the following fields:

#### **Detector and Data Channel**

This field includes the following options:

- **Detector Type** Choose the front/back/aux L/aux R detector module installed on your GC, TRACE 1310 Auxiliary Oven (Valve Oven), or both.
  - Front Select the front detector module (None, FID, ECD, NPD, TCD, FPD, PDD, or GDI) installed on the GC.
  - Back Select the back detector module (None, FID, ECD, NPD, TCD, FPD, PDD, or GDI) installed on the GC.
  - Aux L Select the auxiliary left detector module (None, FID, ECD, NPD, TCD, FPD, PDD, or GDI) installed on the TRACE 1310 Auxiliary Oven module.
  - Aux R Select the auxiliary right detector module (None, FID, ECD, NPD, TCD, FPD, PDD, or GDI) installed on the TRACE 1310 Auxiliary Oven module.

**Note** When GDI is selected, the button **Configure GDI...** is visualized for configuring the GDI detector. See "Configuring a GDI Detector" on page 82.



ATTENTION Chromeleon does not include PDD and GDI modules.

- Make-up Gas Choose the type of make-up gas to use for the front/back/aux L/aux R detector modules.
  - **Front** Choose one: None, Helium, Hydrogen, or Nitrogen.
  - **Back** Choose one: None, Helium, Hydrogen, or Nitrogen.
  - Aux L Choose one: None, Helium, Hydrogen, or Nitrogen.
  - Aux R Choose one: None, Helium, Hydrogen, or Nitrogen.

Note The FPD detector does not have make-up gas so the make-up field is disabled.

- Acquisition Rate (Hz) Select the number of data points to take per second during the acquisition. The maximum acquisition frequency is 10 Hz.
- Channel Name Channels 1 through 4 are the data sources connected to the GC.

#### **Options**

This field includes the following option:

• Line Frequency — Select the AC power frequency (50 Hz or 60 Hz) that your GC is plugged into. The line frequency control indicates allowable scan rate values.

#### **Configuring a GDI Detector**

The GDI electrical interface factory set-up includes three flow restrictors providing a full scale of 50 mL/min of Nitrogen. All three channels are set to **Nitrogen**, and 50 mL/min **full scale** by default.

If a different gas is used, it must be configured, and the full scale also changed accordingly. Use the following gas conversion factors to calculate the new full scale.

He = 1.10	H <sub>2</sub> = 2.20	Air = 0.96	Ar = 0.84	$Ar/CH_4 = 0.86$

To obtain the new full scale, multiply the N<sub>2</sub> full scale (50 mL/min) by the conversion factor.

He = 55.0	H <sub>2</sub> = 110.0	Air = 48.0	Ar = 42.0	$Ar/CH_4 = 43.0$

If higher flow rates or very low flow rates are required, the flow restrictor must be replaced by custom one. When a custom flow restrictor is used, the new full scale must be determined. The module is designed to use 1/4-in. encapsulated porous metal flow restrictors.

**Note** For replacing the encapsulated flow restrictor and determining the new full scale, refer to the sections **Adding a Generic Detector Interface | Replacing the Flow Restrictors** in Chapter 9 of the *TRACE 1300/TRACE 1310 Hardware Manual*.

#### \* To configure a GDI Detector

- 1. In the **Configuration** window select the **Detectors** tab.
- 2. Select the Detector Type: choose GDI. The Configure GDI ... button is visualized.
- 3. Click Configure GDI ...; the GDI Configuration page is visualized.
  - **Gas Type:** Select the type of gas used for the detector gases **Gas1**, **Gas2**, and **Gas3**. Choose one: Air, Hydrogen, Nitrogen, Helium, Argon, or Argon/Methane. Nitrogen is the default gas.
  - **Full-scale flow** Set the full scale value of the restriction installed for each detector gas. Set a value in the range 1-1000 mL/min. Default value is 50 mL/min.
  - Max. detector temperature: Select the maximum detector temperature in the range from 0 °C to 450 °C. The default temperature is 400 °C.
  - ADC full-scale voltage: Select the Analog Digital Converter input full scale voltage. Choose one: 1 V, 5 V, or 10 V. The default value is 1V.

# **Configuration Aux Oven (Valve Oven)Tab**

These controls allow you to configure and use a TRACE 1310 Auxiliary Oven (Valve Oven) coupled with your TRACE 1300/1310 GC.

This tab includes the following fields:

#### Aux Oven (Valve Oven)

- Aux Oven Module (Valve Oven Module)— Select this check box to enable the setting for the TRACE 1310 Auxiliary Oven (Valve Oven). See also "Auxiliary Temperature Page" on page 128.
- Aux Oven Events 1-8 (Valve Oven Events 1-8)— Select this check box to enable Valve type 1-8, and Valve Oven events. See also "Run Table Page" on page 130.
  - Valve type 1-8 Chromeleon CDS only: Defines the type of valves installed into the TRACE 1310 Auxiliary Oven. Select Switching or Sampling.
- Heater 1: Select the component connected to the heater 1 among: Not present, Valve Oven, H. T. Valve Oven, and Aux 3. See also "Auxiliary Temperature Page" on page 128.
- Heater 2: Select the component connected to the heater 2 among: Not present, Aux Col. Oven, Aux 4, and Methanizer.

The temperature limits must be consistent with the configuration as for the auxiliary temperatures. See Table 4.

#### Table 4. Auxiliary Temperature Limits

Туре	Maximum Temperature °C	Available on:	Notes
Transfer Line	400	Aux 1, Aux 2	Primary module, display as Transfer Line.
Valve Oven	200	Aux 3 only	Secondary module (Valve Oven)
High Temperature Valve Oven	300	Aux 3 only	Secondary module (Valve Oven)
Aux Column Oven	250	Aux 4 only	Secondary module (Valve Oven)
Methanizer	400	Aux 4 only	Secondary module (Valve Oven)
Aux Temperature (generic)	400	All Aux 1, 2, 3, 4	Primary and secondary modules, display as Aux Temperature (#)

# **Configuration Auxiliary Temperature Tab**

These controls allow you to configure and use an auxiliary temperature interface.

This tab includes the following field:

#### **Auxiliary Temperature Control**

The available options are:

- Auxiliary Control Module Select this check box to enable the setting for the auxiliary control modules and options installed on your GC. See also "Auxiliary Temperature Page" on page 128.
- External Events 1-8 Select this check box to enable external events. See also "Run Table Page" on page 130.
- Heater 1 Select the component connected to the Heater 1 among: No present, Transfer Line 1, and Aux Heater. See also "Auxiliary Temperature Page" on page 128.
- Heater 2 Select the component connected to the Heater 2 among: No present, Transfer Line 2, and Aux Heater. See also "Auxiliary Temperature Page" on page 128.
- Oven Cryogenics Select this check box if the Oven Cryo option is installed in your GC. See also "Oven Page" on page 87.
- Front/Back Inlet Cryogenics Select this check box if the front/back inlet cryogenic option is installed in your GC.
- **Cryo Type** Select the coolant used by your cryogenic option. Choose one: Liquid Nitrogen or Carbon Dioxide.

# **Configuration Auxiliary Carrier Tab**

These controls allow you to configure and use an Auxiliary Carrier interface.

This menu includes both the Auxiliary Gas modules. One of them will be mounted inside the TRACE 1310 Auxiliary Oven, and the other into the GC. See also Auxiliary Carrier Page.

This tab includes the following field:

#### **Auxiliary Carrier Control**

- Auxiliary Control Module 1 Select this check box to enable the setting for the auxiliary control module and options installed on your GC or TRACE 1310 Auxiliary Oven.
  - Carrier Gas 1 Select the type of the carrier gas in use for the selected auxiliary carrier line 1. The choices are He, H<sub>2</sub>, and N<sub>2</sub>.
  - Back-pressure mode Select the corresponding check box if you want operating in back-pressure mode.
  - Carrier Gas 2 Select the type of the carrier gas in use for the selected auxiliary carrier line 2. The choices are He, H<sub>2</sub>, and N<sub>2</sub>.
  - Back-pressure mode Select the corresponding check box if you want operating in back-pressure mode.
  - **Carrier Gas 3** Select the type of the carrier gas in use for the selected auxiliary carrier line 3. The choices are He,  $H_2$ , and  $N_2$ .
  - Back-pressure mode Select the corresponding check box if you want operating in back-pressure mode.
- Auxiliary Control Module 2 Select this check box to enable the setting for the second auxiliary control module and options installed on your GC or TRACE 1310 Auxiliary Oven.
  - **Carrier Gas 4** Select the type of the carrier gas in use for the selected auxiliary carrier line 4. The choices are He,  $H_2$ , and  $N_2$ .
  - Back-pressure mode Select the corresponding check box if you want operating in back-pressure mode.
  - **Carrier Gas 5** Select the type of the carrier gas in use for the selected auxiliary carrier line 5. The choices are He,  $H_2$ , and  $N_2$ .
  - Back-pressure mode Select the corresponding check box if you want operating in back-pressure mode.
  - Carrier Gas 6 Select the type of the carrier gas in use for the selected auxiliary carrier line 6. The choices are He, H<sub>2</sub>, and N<sub>2</sub>.
  - Back-pressure mode Select the corresponding check box if you want operating in back-pressure mode.

# **Edit Method Parameters**

The parameters to set for the oven, injectors, and detectors, should be set according to the configuration of the GC. After you have configured the TRACE 1300/1310, you are ready to set the controls to run your method.

See the following sections:

- "Oven Page" on page 87
- "S/SL Page" on page 89
- "S/SL Backflush Page" on page 93
- "PTV Page" on page 98
- "PTV Backflush Page" on page 105
- "GSV Page" on page 112
- "Helium Saver (HeS-S/SL) Page" on page 116
- "FID Page" on page 121
- "ECD Page" on page 122
- "NPD Page" on page 123
- "TCD Page" on page 124
- "FPD Page" on page 125
- "PDD Page" on page 126
- "GDI Page" on page 127

# **Oven Page**

This page is the method editor for setting up the Oven parameters. This page includes the following fields:

#### **Oven Temperature Program Graph**

Graphical representations of the oven temperature program including any post run events. The axes are temperature in degrees centigrade and time in minutes. An isothermal run just gives a flat line

#### Ramps

The temperature program comprises an initial isothermal field followed by up to 32 linear ramps. The initial row is displayed by default and cannot be hidden.

The parameters in the program timetable are:

- Ramps buttons Use these buttons to add or remove the number of ramps to use in the oven temperature program. This is the rate in degrees Centigrade per minute the GC oven is ramped up or down from the initial temperature or the previous level's final ramp temperature.
  - Clicking on the 🗣 button automatically adds a ramp level after the last one listed.
  - Clicking on the removes the highest level or the one being displayed on the bottom.
- Rate This field is not available for the initial field of the program. Enter a rate of temperature change in the range of 0.1-125 °C/min.
- Temperature Enter a temperature for the isothermal field of the ramp.
  - The range is from 0-450 °C.
  - The range is from -50 °C to Oven Maximum with carbon dioxide cryogenic installed. Otherwise the lower limit is 0 °C.
  - The range is from -100 °C to Oven Maximum with liquid nitrogen cryogenic installed. Otherwise the lower limit is 0 °C.
- Hold time Enter a time for which the temperature is to be held in the range of 0.00-999.99 min.

#### **Acquisition Time**

Use this field to set the run time. The acquisition time parameters are:

• Oven run time — Select this option if you want to use the time calculated for the oven program as the run time. The value is displayed in the adjacent read-only field.

• Specific time — Select this option if you wish to enter a specific run time. Enter a value in the range 0.01-999.99 min.

## **Options**

Various oven parameters are set.

- Oven On Select this check box to enable the oven control.
- Max temperature Enter a value for the maximum oven temperature in the range 0-450 °C if you are not using cryogenics.

With cryogenics enabled, the lower limit is -50 °C with carbon dioxide and -100 °C with liquid nitrogen.

- This entry controls the maximum temperature values in the temperature program field.
- **Prep-run timeout** Enter a time during which the injection must occur. If this does not happen, the chromatograph returns to the Standby condition. Enter a value in the range 0.00-999.99 min.
- Equilibration time Required for equilibrating the oven temperature after it has been set or modified. Enter a value in the range 0.00-999.99 min.
- Ready delay Set the delay time before the GC enters the Ready to Inject condition. Enter a value in the range of 0–99.9 min. This time must not exceed the **Prep-run Timeout** value.
- Cryogenics enable This option is visualized if Oven cryogenics has been selected in Instrument Configuration. See "Configuration Auxiliary Temperature Tab" on page 84. Select/clear this check box to enable or disable the cryogenic system when it is installed and configured with CO<sub>2</sub> or liquid N<sub>2</sub> as a coolant.
- Cryo threshold This option is visualized if Oven cryogenics has been selected in Instrument Configuration. See "Configuration Auxiliary Temperature Tab" on page 84. Specify the temperature at which the cryogenic system begins to supply the coolant. Enter a value in the range of 40-200 °C.
- Cryo timeout This option is visualized if **Oven cryogenics** has been selected in Instrument Configuration. See "Configuration Auxiliary Temperature Tab" on page 84. Specify the time after which if the GC does not reach the ready condition, it stops the use of the cryogenic stops and remains in NOT ready condition. Re-send the method to restore the function. Enter a value in the range 0.00-999.9 min. The default value is 60.00 minutes.

# S/SL Page

This page is the method editor for the front/back Split/Splitless injector module.

This page includes the following fields:

## S/SL Mode

This parameter enables the fields in this pane.

- Split Enables the Inlet field only. The carrier flow is split in the injection port with the bulk going out the split vent. Use this injection mode when analyzing high concentration or neat samples, or in instances where sensitivity is less important. The split vent remains open all the time. This method yields the sharpest peaks if the split gas is properly mixed.
- Splitless Enables the Inlet and Purge fields. The split vent is closed during the injection to drive most of the sample into the column. The solvent effect is required to refocus the analytes, especially more volatile compounds. You can achieve the solvent effect by keeping the analytical column or guard column slightly below the solvent's boiling point. Splitless times of about 1 minute are typical.
- Splitless w/Surge Enables all fields. Same as Splitless but can also program a surge during an injection. Surge starts at Prep Run and continues until the surge duration time is finished. Surge is further defined in the next group of controls.

## Inlet

The inlet parameters are set in this field which is common to all modes.

- Temperature Select the check box to enable the adjacent field. Enter a value for the inlet temperature in the range of 0-400 °C.
- Split Flow Select the check box to enable the adjacent field. Enter a value in the range of 5-1250 mL/min. The Split Ratio is adjusted automatically. In addition, this value is governed by the initial column flow rate entered on the associated Carrier mode.

If the flow rate is changed, the Split Flow value is adjusted so that the split ratio is maintained. However, if the Split Flow value then falls outside its limits, a warning is generated and you then have to change the entry to a valid number.

- Split Ratio This field is enabled under the following conditions:
  - a. Mode is set to Split.
  - b. The Split Flow check box is selected.
  - c. On the associated Carrier mode, the Flow Mode is set to either Constant Flow or Programmed Flow.

This is the ratio between the split flow and the column flow.

$$SplitRatio = \frac{SplitFlow}{ColumnFlow}$$

Enter a value in the range of 1-12500. The Split Flow entry is adjusted automatically.

• Splitless Time — This field is enabled when the S/SL Mode is set to either Splitless or Splitless w/Surge. Specify the length of time the split valve remains closed after a splitless injection. Enter a value in the range of 0.00-999.99 min.

The timer begins at the start of the run. During this time, most of the sample transfers from the injector to the column. The split vent reopens when the splitless time ends. This occurs in order to remove excess solvent vapor.

#### Surge

This field is enabled when the S/SL Mode is set to Splitless or Splitless w/Surge.

- Surge Pressure The pressure applied during the splitless time to produce a surge of flow in the injector to speed transfer of the sample. It may be used, depending upon the analysis, to sharpen peaks closer to the solvent's boiling point where cold trapping is ineffective and solvent effect is the main refocusing mechanism. Enter a value in the range 5-1000 kPa (0.725-145 psi; 0.05-10 bar).
- Surge Duration This is the time that the surge pressure is maintained. Enter a value in the range 0.00-999.99 min. Typically, set to coincide with the **Splitless time**.

#### **Septum Purge**

This field controls the septum purge for the injector. Septum purge is used to sweep the bottom of the septum reducing contamination from sample's analytes. This prevents carryover from run to run. Septum purge also prevents contamination of the injector from septum bleed.

- Purge Flow This field indicates the flow at which the septum is continuously flushed.
- Constant Septum Purge Select the check box to continuously flush the septum with a
  purge flow.
- Stop Purge Time This field is enabled if the Constant Septum Purge check box is cleared. You can then enter a time at which the septum purge ceases. The range is 0.00-999.99 min.

#### **Carrier Mode**

In this field you select the flow mode for the carrier gas. The choice made here controls the parameters available in the Carrier Flow/ Carrier Pressure and Ramps field.

- **Constant Flow** A single Flow field is available along with a check box to enable or disable it. The graph shows a horizontal line at the flow rate indicated. See Carrier Flow.
- **Constant Pressure** A single Pressure field is available along with a check box to enable or disable it. The graph shows a horizontal line at the pressure indicated. See Carrier Pressure.
- **Programmed Flow** The Flow program fields are displayed. The graph represents the ramps in the program. See Programmed Carrier Flow.
- **Programmed Pressure** The Pressure program fields are displayed. The graph represents the ramps in the program. See Programmed Carrier Pressure.

#### **Carrier Flow**

Use this field to set up flow when the **Constant flow** carrier mode has been selected.

Select the **Flow** check box to enable the Flow field. Enter a value in the range of 0.1-100 mL/min. If you clear the check box, the field is disabled and the graph becomes a horizontal line at a flow rate of zero.

#### **Carrier Pressure**

Use this field to set up pressure when the **Constant pressure** carrier mode has been selected. Select the **Pressure** check box to enable the Pressure field.

Enter a value in the range of 5-1000 kPa (0.725-145 psi; 0.05-10 bar). If you clear the check box, the field is disabled and the graph becomes a horizontal line at zero pressure.

#### **Programmed Carrier Flow**

Use this field to set up a flow program when **Programmed Flow** carrier mode has been selected.

In this mode you can program an initial constant flow rate field followed by up to three ramps.

Select the **Flow enable** check box to enable the flow.

Enable the number of flow ramps required by clicking the 🗳 button.

Conversely, to reduce the number of rows on display, click the 🔀 button as necessary.

The initial row is displayed by default and cannot be hidden.

If you wish to disable the ramps temporarily, clear the **Flow enable** check box. This disables the Flow column and the program is ignored as shown in the graph, which becomes a horizontal line at zero flow rate. The values entered are preserved and may be reactivated by selecting the check box once more.

The parameters in the timetable are:

- **Rate** Enter a value for the rate of flow rate change in the range of 0.001-100 mL/min<sup>2</sup>. This field is not available for the initial field of the program.
- **Flow** Enter a value for the constant flow rate field of the ramp in the range of 0.001-100 mL/min.
- **Hold Time** Enter a value for which the flow rate will be held. The range is 0.00-999.99 min.

#### **Programmed Carrier Pressure**

Use this field to set up a pressure program when **Programmed Pressure** carrier mode has been selected.

In this mode you can program an initial constant pressure rate field followed by up to three ramps.

Select the Pressure enable check box to enable the pressure.

Enable the number of pressure ramps required by clicking on the 🔂 button.

Conversely, to reduce the number of rows on display, click on the 📉 button as necessary.

The initial row is displayed by default and cannot be hidden. If you wish to disable the Pressure ramps temporarily, clear the **Pressure enable** check box. This disables the Pressure column, and the program is ignored as shown in the graph, which becomes a horizontal line at zero pressure. The values entered are preserved and may be reactivated by selecting the check box once more.

The parameters in the timetable are:

- **Rate** Enter a value for the rate of pressure change in the range of 0.01-1000 kPa/min (0.001-145 psi; 0.0001-10 bar). This field is not available for the initial field of the program.
- **Pressure** Enter a value for the constant pressure field of the ramp in the range of 5-1000 kPa (0.725-145 psi; 0.05-10 bar).
- Hold Time Enter a time for which the pressure will be held. The range is 0.00-999.99 min.

#### **Carrier Options**

This field includes the following parameters:

• Vacuum Compensation — Select the check box to enable the vacuum compensation feature. Use this only when the TRACE 1300 is connected to a mass spectrometer detector.

When the check box is not selected, calculations are made for a normal GC detector, which is usually at atmospheric pressure.

• **Gas Saver** — Select the check box to enable the function in this field. Gas saver controls reduce carrier gas consumption, especially when a large split flow is used. It is set to come on at some point well after the injection to conserve gas.

Gas Saver parameters are:

- Gas Saver Flow Enter a value for the gas saver flow rate in the range of 5-500 mL/min.
- Gas Saver Time This is time into the run when the gas saver feature starts up. Enter a value in the range of 0.00-999.99 min.

# S/SL Backflush Page

This page is the method editor for the Front/Back Split/Splitless injector module for Backflush.

This page includes the following fields:

#### S/SL Mode

This parameter enables the fields in this pane.

#### S/SL Mode

This parameter enables the fields in this pane.

- **Split** Enables the Inlet field only. The carrier flow is split in the injection port with the bulk going out the split vent. Use this injection mode when analyzing high concentration or neat samples, or in instances where sensitivity is less important. The split vent remains open all the time. This method yields the sharpest peaks if the split gas is properly mixed.
- Splitless Enables the Inlet and Purge fields. The split vent is closed during the injection to drive most of the sample into the column. The solvent effect is required to refocus the analytes, especially more volatile compounds. You can achieve the solvent effect by keeping the analytical column or guard column slightly below the solvent's boiling point. Splitless times of about 1 minute are typical.
- Splitless w/Surge Enables all fields. Same as Splitless but can also program a surge during an injection. Surge starts at Prep Run and continues until the surge duration time is finished. Surge is further defined in the next group of controls.

## Inlet

The inlet parameters are set in this field which is common to all modes.

- Temperature Select the check box to enable the adjacent field. Enter a value for the inlet temperature in the range of 0-400 °C.
- Split Flow Select the check box to enable the adjacent field. Enter a value in the range of 5-1250 mL/min. The Split Ratio is adjusted automatically. In addition, this value is governed by the initial column flow rate entered on the associated Carrier mode.

If the flow rate is changed, the Split Flow value is adjusted so that the split ratio is maintained. However, if the Split Flow value then falls outside its limits, a warning is generated and you then have to change the entry to a valid number.

- Split Ratio This field is enabled under the following conditions:
  - a. Mode is set to Split.
  - b. The Split Flow check box is selected.
  - c. On the associated Carrier mode, the Flow Mode is set to either Constant Flow or Programmed Flow.

This is the ratio between the split flow and the column flow.

SplitRatio =  $\frac{\text{SplitFlow}}{\text{ColumnFlow}}$ 

Enter a value in the range of 1-12500. The Split Flow entry is adjusted automatically.

• Splitless Time — This field is enabled when the S/SL Mode is set to either Splitless or Splitless w/Surge. Specify the length of time the split valve remains closed after a splitless injection. Enter a value in the range of 0.00-999.99 min.

The timer begins at the start of the run. During this time, most of the sample transfers from the injector to the column. The split vent reopens when the splitless time ends. This occurs in order to remove excess solvent vapor.

## Surge

This field is enabled when the S/SL Mode is set to Splitless or Splitless w/Surge.

- Surge Pressure The pressure applied during the splitless time to produce a surge of flow in the injector to speed transfer of the sample. It may be used, depending upon the analysis, to sharpen peaks closer to the solvent's boiling point where cold trapping is ineffective and solvent effect is the main refocusing mechanism. Enter a value in the range 5-1000 kPa (0.725-145 psi; 0.05-10 bar).
- Surge Duration This is the time that the surge pressure is maintained. Enter a value in the range 0.00-999.99 min. Typically, set to coincide with the **Splitless time**.

#### **Septum Purge**

This field controls the septum purge for the injector. Septum purge is used to sweep the bottom of the septum reducing contamination from sample's analytes. This prevents carryover from run to run. Septum purge also prevents contamination of the injector from septum bleed.

- Purge Flow This field indicates the flow at which the septum is continuously flushed.
- **Constant Septum Purge** Select the check box to continuously flush the septum with a purge flow.
- Stop Purge Time This field is enabled if the Constant Septum Purge check box is cleared. You can then enter a time at which the septum purge ceases. The range is 0.00-999.99 min.

#### **Carrier Mode**

In this field, you select the flow mode for the carrier gas.

The choice made here controls the parameters available in the Carrier Flow/ Carrier Pressure and Ramps field.

- **Constant Flow** A single Flow field is available along with a check box to enable or disable it. The graph shows a horizontal line at the flow rate indicated. See Carrier Flow.
- **Constant Pressure** A single Pressure field is available along with a check box to enable or disable it. The graph shows a horizontal line at the pressure indicated. See Carrier Pressure.
- **Programmed Flow** The Flow program fields are displayed. The graph represents the ramps in the program. See Programmed Carrier Flow.
- **Programmed Pressure** The Pressure program fields are displayed. The graph represents the ramps in the program. See Programmed Carrier Pressure.

## **Carrier Flow**

Use this field to set up flow when the **Constant flow** carrier mode has been selected.

Select the **Flow** check box to enable the Flow field. Enter a value in the range of 0.1-100 mL/min. If you clear the check box, the field is disabled and the graph becomes a horizontal line at a flow rate of zero.

#### **Carrier Pressure**

Use this field to set up pressure when **Constant pressure** carrier mode has been selected.

Select the **Pressure** check box to enable the Pressure field.

Enter a value in the range of 5-1000 kPa (0.725-145 psi; 0.05-10 bar). If you clear the check box, the field is disabled and the graph becomes a horizontal line at zero pressure.

#### **Programmed Carrier Flow**

Use this field to set up a flow program when the **Programmed Flow** carrier mode has been selected.

In this mode you can program an initial constant flow rate field followed by up to three ramps.

Select the Flow enable check box to enable the flow.

Enable the number of flow ramps required by clicking on the 🕏 button.

Conversely, to reduce the number of rows on display, click on the 🔀 button as necessary.

The initial row is displayed by default and cannot be hidden.

If you wish to disable the ramps temporarily, clear the **Flow enable** check box. This disables the Flow column and the program is ignored as shown in the graph, which becomes a horizontal line at zero flow rate. The values entered are preserved and may be reactivated by selecting the check box once more.

The parameters in the timetable are:

- **Rate** Enter a value for the rate of flow rate change in the range of 0.001-100 mL/min<sup>2</sup>. This field is not available for the initial field of the program.
- Flow Enter a value for the constant flow rate field of the ramp in the range of 0.001-100 mL/min.
- **Hold Time** Enter a value for which the flow rate will be held. The range is 0.00-999.99 min.

#### **Programmed Carrier Pressure**

Use this field to set up a pressure program when **Programmed Pressure** carrier mode has been selected.

In this mode you can program an initial constant pressure rate field followed by up to three ramps.

Select the **Pressure enable** check box to enable the pressure.

Enable the number of pressure ramps required by clicking on the 🕏 button.

Conversely, to reduce the number of rows on display, click on the 📉 button as necessary.

The initial row is displayed by default and cannot be hidden. If you wish to disable the Pressure ramps temporarily, clear the **Pressure enable** check box. This disables the Pressure column, and the program is ignored as shown in the graph which, becomes a horizontal line at zero pressure. The values entered are preserved and may be reactivated by selecting the check box once more.

The parameters in the timetable are:

- **Rate** Enter a value for the rate of pressure change in the range 0.01-1000 kPa/min (0.001-145 psi; 0.0001-10 bar). This field is not available for the initial field of the program.
- **Pressure** Enter a value for the constant pressure field of the ramp in the range 5-1000 kPa (0.725-145 psi; 0.05-10 bar).
- Hold Time Enter a time for which the pressure will be held. The range is 0.00-999.99 min.

#### **Carrier Options**

This field includes the following parameters:

- Vacuum Compensation Select the check box to enable the vacuum compensation feature. Use this only when the TRACE 1300 is connected to a mass spectrometer detector. When the check box is not selected, calculations are made for a normal GC detector, which is usually at atmospheric pressure.
- **Gas Saver** Select the check box to enable the function in this field. Gas saver controls reduce carrier gas consumption, especially when a large split flow is used. It is set to come on at some point well after the injection to conserve gas.

Gas Saver parameters are:

- Gas Saver Flow Enter a value for the gas saver flow rate in the range of 5-500 mL/min.
- Gas Saver Time This is time into the run when the gas saver feature starts up. Enter a value in the range of 0.00-999.99 min.

#### **Backflush**

Use this field to set up the Backflush parameters.

- Backflush Enable Select this check box to enable the Backflush field.
- Backflush Start Set the time at which the backflush starts here.
- Backflush Duration Choose between GC Run Time and Specific Time.
  - **GC Run Time** Select this option if you want to use the time calculated for the oven program as the run time.

- Specific Time Select this option if you want to enter a specific run time.
- Duration Enabled when **Specific Time** has been selected. Set the duration of the backflush here in the range of 0.00-999.99 min.

# **PTV Page**

This page is the method editor for the Front/Back Programmable Temperature Vaporizing injector module.

This page includes the following fields:

#### **PTV Mode**

The **Mode** setting controls the availability of the parameters in this pane. Select the appropriate entry for your injector.

All **PTV** injection modes are **programmable ramped temperatures** that you operate at varying temperatures with specified times and temperature increments. Selecting PTV modes activates various controls listed in the **Injections Phases** field.

All **CT** injection modes are **constant temperature** (isothermal) modes you operate at set temperatures and time increments. Selecting CT modes disables all controls in the **Injections Phases** field.

**On-Column** injection mode is selected to use the PTV similarly to an On-Column injector.

The options are:

- **PTV Split** The carrier flow is split in the injection port with the bulk going out the split vent. The split vent remains open all the time.
- **PTV Splitless** Closes the split vent during injection to drive all the sample into the column. Splitless times of about 1 minute are typical.
- PTV Large Volume Eliminates the solvent before the sample enters the column.
- **CT Split** Performs traditional isothermal split operation. The carrier flow is split in the injection port with the bulk going out the split vent. The split vent remains open all the time.
- **CT Splitless** Performs traditional isothermal splitless operation. The split vent is closed during injection to drive all the sample into the column. Splitless times of about 1 minute are typical.
- CT Splitless w/surge Same as Splitless but can also program a surge during an injection. Surge starts at Prep Run and continues until the surge duration time is finished. Surge is further defined in next group of controls.

• **On-Column** — Uses the PTV injector as an On-Column injector. The injector heats with an automatic temperature rate emulating the oven temperature. When using this technique, set the initial oven temperature below the solvent boiling point.

#### Inlet

The inlet parameters are set in this field.

• Temperature — Select the check box to enable the adjacent field. If the cryogenic option is present, enter a value for the inlet temperature in the range of - 50 to 450 °C with carbon dioxide, and from -100 to 450 °C with liquid nitrogen. Otherwise the lower limit is 0 °C. For non-cryogenic use, this temperature is usually set just above the solvent boiling point.

Optimum temperature for an analytical method varies with the method and sample requirements. The entry also has a check box for on/off values, so that the temperature setting may be turned off without affecting the setpoint (usually used for troubleshooting purposes).

• Split Flow — Select the check box to enable the adjacent field. Enter a value in the range of 5-1250 mL/min. The Split Ratio is adjusted automatically. In addition, this value is governed by the initial column flow rate entered on the associated Carrier mode.

If the flow rate is changed, the Split Flow value is adjusted so that the split ratio is maintained. However, if the Split Flow value then falls outside its limits, a warning is generated and you must change the entry to a valid number.

- Split Ratio This field is enabled under the following conditions:
  - a. Mode is set to PTV Split or CT Split.
  - b. The Split Flow check box is selected.
  - c. On the associated Carrier mode, Flow Mode is set to either Constant Flow or Programmed Flow.

This is the ratio between the split flow and the column flow.

SplitRatio = 
$$\frac{\text{SplitFlow}}{\text{ColumnFlow}}$$

Enter a value in the range of 1-12500. The Split Flow entry is adjusted automatically.

• Splitless Time — This field is enabled when the **Mode** is set to either **Splitless** or **Splitless w/Surge**. Specify the length of time the split valve remains closed after a splitless injection. The timer begins at the start of the run. During this time, most of the sample transfers from the injector to the column. The split vent reopens when the splitless time ends. This occurs in order to remove excess solvent vapor. Enter a value in the range of 0.00-999.99 min.

This field is disabled when the Mode is set to either PTV Split or CT Split. It is the time for which the split valve is closed. Enter a value in the range of 0.00-999.99 min.

## Surge

This field is enabled when the Mode is set to CT Splitless w/Surge.

- Surge Pressure The CT Splitless w/Surge mode allows a higher inlet pressure to be applied during injection. This serves to reduce the volume of the vapor cloud created when the injected sample is vaporized, and may improve the resolution of the separated analytes. Enter a value in the range of 5-1000 kPa (0.725-145 psi; 0.05-10 bar).
- Surge Duration This is the amount of time that the inlet pressure will be increased, during which the analytes will transfer more rapidly to the column.Enter a value in the range of 0.00-999.99 min.

### **Septum Purge**

This field controls the septum purge for the injector. Septum purge is used to sweep the bottom of the septum to reduce contamination from the sample's analytes. This prevents carryover from run to run. Septum purge also prevents contamination of the injector from septum bleed.

- Purge Flow This field indicates the flow at which the septum is continuously flushed.
- **Constant Septum Purge** Select the check box to continuously flush the septum with a purge flow.
- Stop Purge Time This field is enabled if the Constant Septum Purge check box is cleared. You can then enter a time at which the septum purge ceases. The range is 0.00-999.99 min.

## **Carrier Mode**

In this field, you select the flow mode for the carrier gas.

The choice made here controls the parameters available in the Carrier Flow/ Carrier Pressure and Ramps field.

- **Constant Flow** A single Flow field is available along with a check box to enable or disable it. The graph shows a horizontal line at the flow rate indicated. See Carrier Flow.
- **Constant Pressure** A single Pressure field is available along with a check box to enable or disable it. The graph shows a horizontal line at the pressure indicated. See Carrier Pressure.
- **Programmed Flow** The Flow program fields are displayed. The graph represents the ramps in the program. See Programmed Carrier Flow.
- **Programmed Pressure** The Pressure program fields are displayed. The graph represents the ramps in the program. See Programmed Carrier Pressure.

#### **Carrier Flow**

Use this field to set up flow when the **Constant flow** carrier mode has been selected.

Select the **Flow** check box to enable the Flow field.

Enter a value in the range of 0.1-100 mL/min. If you clear the check box, the field is disabled and the graph becomes a horizontal line at a flow rate of zero.

#### **Carrier Pressure**

Use this field to set up pressure when the Constant pressure carrier mode has been selected.

Select the **Pressure** check box to enable the Pressure field.

Enter a value in the range of 5-1000 kPa (0.725-145 psi; 0.05-10 bar). If you clear the check box, the field is disabled, and the graph becomes a horizontal line at zero pressure.

### **Programmed Carrier Flow**

Use this field to set up flow program when the **Programmed Flow** carrier mode has been selected.

In this mode you can program an initial constant flow rate field followed by up to three ramps.

Select the Flow enable check box to enable the flow.

Enable the number of flow ramps required by clicking on the 🗘 button.

Conversely, to reduce the number of rows on display, click on the 🔀 button as necessary.

The initial row is displayed by default and cannot be hidden.

If you wish to disable the ramps temporarily, clear the **Flow enable** check box. This disables the Flow column and the program is ignored as shown in the graph, which becomes a horizontal line at zero flow rate. The values entered are preserved and may be reactivated by selecting the check box once more.

The parameters in the timetable are:

- **Rate** Enter a value for the rate of flow rate change in the range of 0.001-100 mL/min<sup>2</sup>. This field is not available for the initial field of the program.
- Flow Enter a value for the constant flow rate field of the ramp in the range 0.001-100 mL/min.
- **Hold Time** Enter a value for which the flow rate will be held. The range is 0.00-999.99 min.

## **Programmed Carrier Pressure**

Use this field to set up pressure program when **Programmed Pressure** carrier mode has been selected.

In this mode you can program an initial constant pressure rate field followed by up to three ramps.

Select the Pressure enable check box to enable the pressure.

Enable the number of pressure ramps required by clicking on the 🔁 button.

Conversely, to reduce the number of rows on display, click on the 📉 button as necessary.

The initial row is displayed by default and cannot be hidden. If you wish to disable the Pressure ramps temporarily, clear the **Pressure enable** check box. This disables the Pressure column and the program is ignored as shown in the graph, which becomes a horizontal line at zero pressure. The values entered are preserved and may be reactivated by selecting the check box once more.

The parameters in the timetable are:

- **Rate** Enter a value for the rate of pressure change in the range of 0.01-1000 kPa/min (0.001-145 psi; 0.0001-10 bar). This field is not available for the initial field of the program.
- **Pressure** Enter a value for the constant pressure field of the ramp in the range of 5-1000 kPa (0.725-145 psi; 0.05-10 bar).
- Hold Time Enter a time for which the pressure will be held. The range is 0.00-999.99 min.

#### **Carrier Options**

This field includes the following parameters:

- Vacuum Compensation Select the check box to enable the vacuum compensation feature. Use this only when the TRACE 1300 is connected to a mass spectrometer detector. When the check box is not selected, calculations are made for a normal GC detector, which is usually at atmospheric pressure.
- **Gas Saver** Select the check box to enable the function in this field. Gas saver controls reduce carrier gas consumption, especially when a large split flow is used. It is set to come on at some point well after the injection to conserve gas.

Gas Saver parameters are:

 Gas Saver Flow — Enter a value for the gas saver flow rate in the range of 5-500 mL/min. - Gas Saver Time — This is time into the run when the gas saver feature starts up. Enter a value in the range of 0.00-999.99 min.

#### **Injection Phases**

This field is enabled for PTV mode options only. The fields are activated by selections made in the **Options** field. Further restrictions apply. These are described as necessary.

There are four phases: Injection, Evaporation, Transfer and Cleaning.

- Injection Use this control for the injection's ramped pressure. Available only with PTV Splitless or PTV Large Volume mode.
- Evaporation It is enabled when Evaporation phase check box is selected. This group of controls allows you to specify solvent evaporation controls: pressure (PTV Splitless only), rate, temperature, and time in minutes. Set the solvent evaporation temperature; set the rate to reach the solvent evaporation temperature.
- Transfer This group of controls allows you to specify controls for the sample transfer into the column: Pressure (PTV Splitless only), rate, temperature, and time in minutes. Specify the pressure if available. Set the rate in °C/s to reach the sample transfer temperature. Set the temperature for sample transfer into the column. Set the time in minutes for the transfer temperature to be maintained.
- Cleaning It is enabled when Cleaning phase check box is selected. Set the rate necessary to reach the cleaning temperature. Set the injector cleaning temperature. Set the time in minutes the cleaning temperature must be maintained.
- Evaporation Phase Select this check box to enable the Evaporation phase parameters in the Injection Phases field.
- Cleaning Phase Select this check box to enable the Cleaning phase parameters in the Injection Phases field.
- **Ramped Pressure** Select this check box to enable the Pressure parameters in the Injection Phases field. This is enabled only when Mode is set to PTV Splitless.
- Transfer temp. delay It is active only when PTV Large Volume mode is set. It delays the temperature ramp start after evaporation phase. Enter a value in the range of 0.00 to 999.99
- Post-cycle temperature Choose one among Turn Off, Cool Down, Maintain as required
- **Pressure** This column is enabled only when Mode is PTV Splitless and the Ramped Pressure check box is selected. Enter a value in the range of 5-1000 kPa (0.725-145 psi; 0.05-10 bar). This parameter does not apply to the Cleaning phase.
- Rate This column is enabled only when the Evaporation Phase check box is selected. Enter a value in the range of 0.1-14.5 °C/min. This parameter does not apply to the Injection phase.

- Temp This is the temperature of the phase. Enter a value in the range of 0-450°C (from 50 to 450 °C with carbon dioxide, and from -100 to 450 °C with liquid nitrogen).
- Time This is the phase temperature holding time. Enter a value in the range 0.00-999.99 min.
- Flow This parameter applies only to the Injection and Cleaning phases. For the Injection phase, the Mode must be set to PTV Large Volume. For the Cleaning phase, the Cleaning Phase check box must be selected. Enter a value in the range of 5-1250 mL/min.

#### **Show Graph**

To view the PTV phases dialog box, click **Show Graph**. This is a read-only, graphical display of changes in temperature, flow rate, and valve state with time. You can use the arrow buttons in the bottom left-hand corner to expand the time scale and view the early phases in more detail. The left-hand button expands the scale while the right-hand one contracts it. The graph cannot be scrolled in expanded mode.

#### **Cryogenics**

This field is visualized if **Front/Back Inlet cryogenics** has been enabled in Instrument Configuration. See "Configuration Auxiliary Temperature Tab" on page 84.

The Cryogenics parameters are:

- Cryogenics enable Select this check box to enable the cryogenic system.
- Cool during Select when cooling the instrument. Choose between Prep-Run and Post -Run,
- Cryo threshold Specify the temperature at which the cryo system begins to supply the coolant. Enter a value in the range of 40-200 °C.
- Cryo timeout Enter the time at which the cryo system will be disabled. Enter a value in the range of 0–30 min.

# **PTV Backflush Page**

This page is the method editor for the Front/Back Programmable Temperature Vaporizing injector module for Backflush.

#### **PTV Mode**

The **Mode** setting controls the availability of the parameters in this pane. Select the appropriate entry for your injector.

All **PTV** injection modes are **programmable ramped temperatures** that you operate at varying temperatures with specified times and temperature increments. Selecting PTV modes activates various controls listed in the **Injections Phases** field.

All **CT** injection modes are **constant temperature** (isothermal) modes you operate at set temperatures and time increments. Selecting CT modes disables all controls in the **Injections Phases** field.

The options are:

- **PTV Split** The carrier flow is split in the injection port with the bulk going out the split vent. The split vent remains open all the time.
- **PTV Splitless** Closes the split vent during injection to drive all the sample into the column. Splitless times of about 1 minute are typical.
- PTV Large Volume Eliminates the solvent before the sample enters the column.
- **CT Split** Performs traditional isothermal split operation. The carrier flow is split in the injection port with the bulk going out the split vent. The split vent remains open all the time.
- **CT Splitless** Performs traditional isothermal splitless operation. The split vent is closed during injection to drive all the sample into the column. Splitless times of about 1 minute are typical.
- CT Splitless w/surge Same as Splitless but can also program a surge during an injection. Surge starts at Prep Run and continues until the surge duration time is finished. Surge is further defined in next group of controls.

#### Inlet

The inlet parameters are set in this field.

• Temperature — Select the check box to enable the adjacent field. If the cryogenic option is present, enter a value for the inlet temperature in the range of - 50 to 450 °C with carbon dioxide, and from -100 to 450 °C with liquid nitrogen. Otherwise the lower limit is 0 °C. For non-cryogenic use, this temperature is usually set just above the solvent boiling point.

Optimum temperature for an analytical method varies with the method and sample requirements. The entry also has a check box for on/off values, so that the temperature setting may be turned off without affecting the setpoint (usually used for troubleshooting purposes).

• Split Flow — Select the check box to enable the adjacent field. Enter a value in the range of 5-1250 mL/min. The **Split Ratio** is adjusted automatically. In addition, this value is governed by the initial column flow rate entered on the associated **Carrier mode**.

If the flow rate is changed, the Split Flow value is adjusted so that the split ratio is maintained. However, if the Split Flow value then falls outside its limits, a warning is generated and you must change the entry to a valid number.

- Split Ratio This field is enabled under the following conditions:
  - a. Mode is set to PTV Split or CT Split.
  - b. The Split Flow check box is selected.
  - c. On the associated Carrier mode, Flow Mode is set to either Constant Flow or Programmed Flow.

This is the ratio between the split flow and the column flow.

$$SplitRatio = \frac{SplitFlow}{ColumnFlow}$$

Enter a value in the range of 1-12500. The Split Flow entry is adjusted automatically.

• Splitless Time — This field is enabled when the **Mode** is set to either **Splitless** or **Splitless w/Surge**. Specify the length of time the split valve remains closed after a splitless injection. The timer begins at the start of the run. During this time, most of the sample transfers from the injector to the column. The split vent reopens when the splitless time ends. This occurs in order to remove excess solvent vapor. Enter a value in the range of 0.00-999.99 min.

This field is disabled when the Mode is set to either PTV Split or CT Split. It is the time for which the split valve is closed. Enter a value in the range of 0.00-999.99 min.

## Surge

This field is enabled when the Mode is set to CT Splitless w/Surge.

- Surge Pressure The **CT Splitless w/Surge** mode allows a higher inlet pressure to be applied during injection. This serves to reduce the volume of the vapor cloud created when the injected sample is vaporized, and may improve the resolution of the separated analytes. Enter a value in the range of 5-1000 kPa (0.725-145 psi; 0.05-10 bar).
- Surge Duration This is the amount of time that the inlet pressure will be increased, during which the analytes will transfer more rapidly to the column.Enter a value in the range of 0.00-999.99 min.

#### **Septum Purge**

This field controls the septum purge for the injector. Septum purge is used to sweep the bottom of the septum to reduce contamination from the sample's analytes. This prevents carryover from run to run. Septum purge also prevents contamination of the injector from septum bleed.

- Purge Flow This field indicates the flow at which the septum is continuously flushed.
- **Constant Septum Purge** Select the check box to continuously flush the septum with a purge flow.
- Stop Purge Time This field is enabled if the Constant Septum Purge check box is cleared. You can then enter a time at which the septum purge ceases. The range is 0.00-999.99 min.

#### **Carrier Mode**

In this field, you select the flow mode for the carrier gas.

The choice made here controls the parameters available in the Carrier Flow/ Carrier Pressure and Ramps field.

- **Constant Flow** A single Flow field is available along with a check box to enable or disable it. The graph shows a horizontal line at the flow rate indicated. See Carrier Flow.
- **Constant Pressure** A single Pressure field is available along with a check box to enable or disable it. The graph shows a horizontal line at the pressure indicated. See Carrier Pressure.
- **Programmed Flow** The Flow program fields are displayed. The graph represents the ramps in the program. See Programmed Carrier Flow.
- **Programmed Pressure** The Pressure program fields are displayed. The graph represents the ramps in the program. See Programmed Carrier Pressure.

## **Carrier Flow**

Use this field to set up flow when the **Constant flow** carrier mode has been selected.

Select the **Flow** check box to enable the Flow field. Enter a value in the range of 0.1-100 mL/min. If you clear the check box, the field is disabled and the graph becomes a horizontal line at a flow rate of zero.

#### **Carrier Pressure**

Use this field to set up pressure when the Constant pressure carrier mode has been selected.

Select the **Pressure** check box to enable the Pressure field.

Enter a value in the range of 5-1000 kPa (0.725-145 psi; 0.05-10 bar). If you clear the check box, the field is disabled and the graph becomes a horizontal line at zero pressure.

#### **Programmed Carrier Flow**

Use this field to set up flow program when the **Programmed Flow** carrier mode has been selected.

In this mode you can program an initial constant flow rate field followed by up to three ramps.

Select the Flow enable check box to enable the flow.

Enable the number of flow ramps required by clicking on the 🕏 button.

Conversely, to reduce the number of rows on display, click on the 🔀 button as necessary.

The initial row is displayed by default and cannot be hidden.

If you wish to disable the ramps temporarily, clear the **Flow enable** check box. This disables the Flow column and the program is ignored as shown in the graph, which becomes a horizontal line at zero flow rate. The values entered are preserved and may be reactivated by selecting the check box once more.

The parameters in the timetable are:

- **Rate** Enter a value for the rate of flow rate change in the range 0.001-100 mL/min<sup>2</sup>. This field is not available for the initial field of the program.
- **Flow** Enter a value for the constant flow rate field of the ramp in the range 0.001-100 mL/min.
- **Hold Time** Enter a value for which the flow rate will be held. The range is 0.00-999.99 min.

#### **Programmed Carrier Pressure**

Use this field to set up pressure program when the **Programmed Pressure** carrier mode has been selected.

In this mode you can program an initial constant pressure rate field followed by up to three ramps.

Select the **Pressure enable** check box to enable the pressure.

Enable the number of pressure ramps required by clicking on the 🗳 button.

Conversely, to reduce the number of rows on display, click on the 📉 button as necessary.

The initial row is displayed by default and cannot be hidden. If you wish to disable the Pressure ramps temporarily, clear the **Pressure enable** check box. This disables the Pressure column and the program is ignored as shown in the graph, which becomes a horizontal line at zero pressure. The values entered are preserved and may be reactivated by selecting the check box once more.

The parameters in the timetable are:

- **Rate** Enter a value for the rate of pressure change in the range of 0.01-1000 kPa/min (0.001-145 psi; 0.0001-10 bar). This field is not available for the initial field of the program.
- **Pressure** Enter a value for the constant pressure field of the ramp in the range of 5-1000 kPa (0.725-145 psi; 0.05-10 bar).
- **Hold Time** Enter a time for which the pressure will be held. The range is 0.00-999.99 min.

#### **Carrier Options**

This field includes the following parameters:

- Vacuum Compensation Select the check box to enable the vacuum compensation feature. Use this only when the TRACE 1300 is connected to a mass spectrometer detector. When the check box is not selected, calculations are made for a normal GC detector, which is usually at atmospheric pressure.
- **Gas Saver** Select the check box to enable the function in this field. Gas saver controls reduce carrier gas consumption, especially when a large split flow is used. It is set to come on at some point well after the injection to conserve gas.

Gas Saver parameters are:

- Gas Saver Flow Enter a value for the gas saver flow rate in the range of 5-500 mL/min.
- Gas Saver Time This is time into the run when the gas saver feature starts up. Enter a value in the range of 0.00-999.99 min.

#### **Injection Phases**

This field is enabled for PTV mode options only. The fields are activated by selections made in the **Options** field. Further restrictions apply. These are described as necessary.

There are four phases: Injection, Evaporation, Transfer and Cleaning.

• Injection — Use this control for the injection's ramped pressure. Available only with PTV Splitless or PTV Large Volume mode.

- Evaporation It is enabled when Evaporation phase check box is selected. This group of controls allows you to specify solvent evaporation controls: pressure (PTV Splitless only), rate, temperature, and time in minutes. Set the solvent evaporation temperature; set the rate to reach the solvent evaporation temperature.
- Transfer This group of controls allows you to specify controls for the sample transfer into the column: Pressure (PTV Splitless only), rate, temperature, and time in minutes. Specify the pressure if available. Set the rate in °C/s to reach the sample transfer temperature. Set the temperature for sample transfer into the column. Set the time in minutes for the transfer temperature to be maintained.
- Cleaning It is enabled when Cleaning phase check box is selected. Set the rate necessary to reach the cleaning temperature. Set the injector cleaning temperature. Set the time in minutes the cleaning temperature must be maintained.
- Evaporation Phase Select this check box to enable the Evaporation phase parameters in the Injection Phases field.
- Cleaning Phase Select this check box to enable the Cleaning phase parameters in the Injection Phases field.
- Ramped Pressure Select this check box to enable the Pressure parameters in the Injection Phases field. This is enabled only when Mode is set to PTV Splitless.
- Transfer temp. delay It is active only when PTV Large Volume mode is set. It delays the temperature ramp start after evaporation phase. Enter a value in the range of 0.00 to 999.99
- Post-cycle temperature Choose one among Turn Off, Cool Down, Maintain as required
- **Pressure** This column is enabled only when Mode is PTV Splitless and the Ramped Pressure check box is selected. Enter a value in the range of 5-1000 kPa (0.725-145 psi; 0.05-10 bar). This parameter does not apply to the Cleaning phase.
- **Rate** This column is enabled only when the Evaporation Phase check box is selected. Enter a value in the range of 0.1-14.5 °C/min. This parameter does not apply to the Injection phase.
- Temp This is the temperature of the phase. Enter a value in the range of 0-450°C (from 50 to 450 °C with carbon dioxide, and from -100 to 450 °C with liquid nitrogen).
- Time This is the phase temperature holding time. Enter a value in the range 0.00-999.99 min.
- Flow This parameter applies only to the Injection and Cleaning phases. For the Injection phase, the Mode must be set to **PTV Large Volume**. For the Cleaning phase, the Cleaning Phase check box must be selected. Enter a value in the range of 5-1250 mL/min.

#### **Show Graph**

To view the PTV phases dialog box, click **Show Graph**. This is a read-only, graphical display of changes in temperature, flow rate, and valve state with time. You can use the arrow buttons in the bottom left-hand corner to expand the time scale and view the early phases in more detail. The left-hand button expands the scale while the right-hand one contracts it. The graph cannot be scrolled in expanded mode.

#### Backflush

This field is visualized when CT Split, CT Splitless or CT Splitless w Surge mode has been selected. In the PTV modes the backflush can be enabled during the injection, evaporation, and cleaning phases depending on the mode chosen. Use this field to set up the Backflush parameters.

- Backflush Enable Select this check box to enable the Backflush field.
- Backflush Start Set the time at which the backflush starts here.
- Backflush Duration Choose between GC Run Time and Specific Time.
  - GC Run Time Select this option if you want to use the time calculated for the oven program as the run time.
  - **Specific Time** Select this option if you want to enter a specific run time.
- Duration Enabled when **Specific Time** has been selected. Set the duration of the backflush here in the range of 0.00-999.99 min.

#### **Cryogenics**

This field is visualized if **Front/Back Inlet cryogenics** has been enabled in Instrument Configuration. See "Configuration Auxiliary Temperature Tab" on page 84.

The Cryogenics parameters are:

- Cryogenics enable Select this check box to enable the cryogenic system.
- Cool during Select when cooling the instrument. Choose between Prep-Run and Post -Run,
- Cryo threshold Specify the temperature at which the cryo system begins to supply the coolant. Enter a value in the range of 40-200 °C.
- Cryo timeout Enter the time at which the cryo system will be disabled. Enter a value in the range of 0–30 min.

# **GSV** Page

This page is the method editor for the Front/Back Gas Sampling Valve (GSV) module.

This page includes the following fields:

## S/SL Mode

This parameter enables the fields in this pane.

• **Split** — This option enables the Inlet field only. The carrier flow is split in the injection port with the bulk going out the split vent. Use this injection mode when analyzing high concentration or neat samples, or in instances where sensitivity is less important. The split vent remains open all the time. This method yields the sharpest peaks if the split gas is properly mixed.



**IMPORTANT** When **Split** mode is selected the Gas Sampling Valve parameters in the **Valve** field are enabled. See "Valve" on page 113.

• Splitless — This option enables the Inlet and Purge fields. The split vent is closed during the injection to drive most of the sample into the column. The solvent effect is required to refocus the analytes, especially more volatile compounds. You can achieve the solvent effect by keeping the analytical column or guard column slightly below the solvent's boiling point. Splitless times of about 1 minute are typical.



**IMPORTANT** When **Splitless** mode is selected the Gas Sampling Valve parameters in the **Valve** field are disabled. In this case the **Sampling time** coincides with the **Splitless Time**. See "Valve" on page 113.

• Splitless w/Surge — This option enables all fields. Same as Splitless but can also program a surge during an injection. Surge starts at Prep Run and continues until the surge duration time is finished. Surge is further defined in the next group of controls.



**IMPORTANT** When **Splitless w/Surge** mode is selected the Gas Sampling Valve parameters in the **Valve** field are disabled. In this case the **Sampling time** coincides with the **Splitless Time**. See "Valve" on page 113.

## Inlet

The inlet parameters are set in this field which is common to all modes.

• Temperature — Select the check box to enable the adjacent field. Enter a value for the gas sampling valve temperature in the range 0-150 °C.

• Split Flow — Select the check box to enable the adjacent field. Enter a value in the range of 5-1250 mL/min. The Split Ratio is adjusted automatically. In addition, this value is governed by the initial column flow rate entered on the associated Carrier mode.

If the flow rate is changed, the Split Flow value is adjusted so that the split ratio is maintained. However, if the Split Flow value then falls outside its limits, a warning is generated and you then have to change the entry to a valid number.

- Split Ratio This field is enabled under the following conditions:
  - a. Mode is set to Split.
  - b. The Split Flow check box is selected.
  - c. On the associated Carrier mode, the Flow Mode is set to either Constant Flow or Programmed Flow.

This is the ratio between the split flow and the column flow.

$$SplitRatio = \frac{SplitFlow}{ColumnFlow}$$

Enter a value in the range of 1-12500. The Split Flow entry is adjusted automatically.

• Splitless Time — This field is enabled when the S/SL Mode is set to either Splitless or Splitless w/Surge. The splitless time coincides with the injection duration parameter of the gas sampling valve. Enter a value in the range of 0.00-999.99 min. The timer begins at the start of the run. During this time, most of the sample transfers from the sample loop to the column. The split vent reopens when the splitless time ends. This occurs in order to remove excess solvent vapor.

#### Surge

This field is enabled when the S/SL Mode is set to Splitless or Splitless w/Surge.

- Surge Pressure The pressure applied during the splitless time to produce a surge of flow in the injector to speed transfer of the sample. It may be used, depending upon the analysis, to sharpen peaks closer to the solvent's boiling point where cold trapping is ineffective and solvent effect is the main refocusing mechanism. Enter a value in the range of 5-1000 kPa (0.725-145 psi; 0.05-10 bar).
- Surge Duration This is the time for which the surge pressure is maintained. Enter a value in the range of 0.00-999.99 min. Typically, set to coincide with the **Splitless time**.

#### Valve

This field is enabled when the **S/SL Mode** is set to **Split** and allows the injection with the gas sampling valve.

The field is disabled when **S/SL Mode** set to **Splitless** or **Splitless w/Surge**; in this case the Sampling time coincides with the Splitless time.

- Injection start time Defines the time at which the injection must begin.
- Injection duration Defines the time the sampling valve must be maintained on injection position.

### **Carrier Mode**

In this field, you select the flow mode for the carrier gas.

The choice made here controls the parameters available in the Carrier Flow/Carrier Pressure and Ramps field.

- **Constant Flow** A single Flow field is available along with a check box to enable or disable it. The graph shows a horizontal line at the flow rate indicated. See Carrier Flow.
- **Constant Pressure** A single Pressure field is available along with a check box to enable or disable it. The graph shows a horizontal line at the pressure indicated. See Carrier Pressure.
- **Programmed Flow** The Flow program fields are displayed. The graph represents the ramps in the program. See Programmed Carrier Flow.
- **Programmed Pressure** The Pressure program fields are displayed. The graph represents the ramps in the program. See Programmed Carrier Pressure.

#### **Carrier Flow**

Use this field to set up flow when the **Constant flow** carrier mode has been selected.

Select the **Flow** check box to enable the Flow field. Enter a value in the range of 0.1-100 mL/min. If you clear the check box, the field is disabled and the graph becomes a horizontal line at a flow rate of zero.

#### **Carrier Pressure**

Use this field to set up pressure when **Constant pressure** carrier mode has been selected.

Select the **Pressure** check box to enable the Pressure field. Enter a value in the range of 5-1000 kPa (0.725-145 psi; 0.05-10 bar). If you clear the check box, the field is disabled and the graph becomes a horizontal line at zero pressure.

#### **Programmed Carrier Flow**

Use this field to set up a flow program when the **Programmed Flow** carrier mode has been selected.

In this mode you can program an initial constant flow rate field followed by up to three ramps.

Select the Flow enable check box to enable the flow.

Enable the number of flow ramps required by clicking on the 🗳 button.

Conversely, to reduce the number of rows on display, click on the 📉 button as necessary.

The initial row is displayed by default and cannot be hidden.

If you wish to disable the ramps temporarily, clear the **Flow enable** check box. This disables the Flow column and the program is ignored as shown in the graph, which becomes a horizontal line at zero flow rate. The values entered are preserved and may be reactivated by selecting the check box once more.

The parameters in the timetable are:

- **Rate** Enter a value for the rate of flow rate change in the range of 0.001-100 mL/min<sup>2</sup>. This field is not available for the initial field of the program.
- Flow Enter a value for the constant flow rate field of the ramp in the range of 0.001-100 mL/min.
- **Hold Time** Enter a value for which the flow rate will be held. The range is 0.00-999.99 min.

#### **Programmed Carrier Pressure**

Use this field to set up a pressure program when **Programmed Pressure** carrier mode has been selected.

In this mode you can program an initial constant pressure rate field followed by up to three ramps.

Select the **Pressure enable** check box to enable the pressure.

Enable the number of pressure ramps required by clicking on the 🕈 button.

Conversely, to reduce the number of rows on display, click on the 🔀 button as necessary.

The initial row is displayed by default and cannot be hidden. If you wish to disable the Pressure ramps temporarily, clear the **Pressure enable** check box. This disables the Pressure column, and the program is ignored as shown in the graph which, becomes a horizontal line at zero pressure. The values entered are preserved and may be reactivated by selecting the check box once more.

The parameters in the timetable are:

• **Rate** — Enter a value for the rate of pressure change in the range 0.01-1000 kPa/min (0.001-145 psi; 0.0001-10 bar). This field is not available for the initial field of the program.

- **Pressure** Enter a value for the constant pressure field of the ramp in the range 5-1000 kPa (0.725-145 psi; 0.05-10 bar).
- Hold Time Enter a time for which the pressure will be held. The range is 0.00-999.99 min.

#### **Carrier Options**

This field includes the following parameters:

- Vacuum Compensation Select the check box to enable the vacuum compensation feature. Use this only when the TRACE 1300 is connected to a mass spectrometer detector. When the check box is not selected, calculations are made for a normal GC detector, which is usually at atmospheric pressure.
- **Gas Saver** Select the check box to enable the function in this field. Gas saver controls reduce carrier gas consumption, especially when a large split flow is used. It is set to come on at some point well after the injection to conserve gas.

Gas Saver parameters are:

- Gas Saver Flow Enter a value for the gas saver flow rate in the range of 5-500 mL/min.
- Gas Saver Time This is time into the run when the gas saver feature starts up. Enter a value in the range of 0.00-999.99 min.

#### **Backflush**

Use this field to set up the Backflush parameters.

- Backflush Enable Select this check box to enable the Backflush field.
- Backflush Start Set the time at which the backflush starts here.
- Backflush Duration Choose between GC Run Time and Specific Time.
  - **GC Run Time** Select this option if you want to use the time calculated for the oven program as the run time.
  - **Specific Time** Select this option if you want to enter a specific run time.
- Duration Enabled when **Specific Time** has been selected. Set the duration of the backflush here in the range of 0.00-999.99 min.

# Helium Saver (HeS-S/SL) Page

This page is the method editor for the front/back Instant Connect Helium Saver Injector (HeS-S/SL) module. S/SL Mode

This parameter enables the fields in this pane.

- Split This option enables the Inlet field only. The carrier flow is split in the injection port with the bulk going out the split vent. Use this injection mode when analyzing high concentration or neat samples, or in instances where sensitivity is less important. The split vent remains open all the time. This method yields the sharpest peaks if the split gas is properly mixed.
- **Splitless** This option enables the Inlet and Purge fields. The split vent is closed during the injection to drive most of the sample into the column. The solvent effect is required to refocus the analytes, especially more volatile compounds. You can achieve the solvent effect by keeping the analytical column or guard column slightly below the solvent's boiling point. Splitless times of about 1 minute are typical.
- Splitless w/Surge This option enables all fields. Same as Splitless but can also program a surge during an injection. Surge starts at Prep Run and continues until the surge duration time is finished. Surge is further defined in the next group of controls.

#### Inlet

The inlet parameters are set in this field which is common to all modes.

- Temperature Select the check box to enable the adjacent field. Enter a value for the gas sampling valve temperature in the range 0-150 °C.
- Split Flow Select the check box to enable the adjacent field. Enter a value in the range of 5-1250 mL/min. The Split Ratio is adjusted automatically. In addition, this value is governed by the initial column flow rate entered on the associated Carrier mode.

If the flow rate is changed, the Split Flow value is adjusted so that the split ratio is maintained. However, if the Split Flow value then falls outside its limits, a warning is generated and you then have to change the entry to a valid number.

- Split Ratio This field is enabled under the following conditions:
  - a. Mode is set to Split.
  - b. The Split Flow check box is selected.
  - c. On the associated Carrier mode, the Flow Mode is set to either Constant Flow or Programmed Flow.

This is the ratio between the split flow and the column flow.

SplitRatio = 
$$\frac{\text{SplitFlow}}{\text{ColumnFlow}}$$

Enter a value in the range of 1-12500. The Split Flow entry is adjusted automatically.

• Splitless Time — This field is enabled when the S/SL Mode is set to either Splitless or Splitless w/Surge. The splitless time coincides with the injection duration parameter of the gas sampling valve. Enter a value in the range of 0.00-999.99 min. The timer begins at the start of the run. During this time, most of the sample transfers from the sample loop to the column. The split vent reopens when the splitless time ends. This occurs in order to remove excess solvent vapor.

#### Surge

This field is enabled when the S/SL Mode is set to Splitless or Splitless w/Surge.

- Surge Pressure The pressure applied during the splitless time to produce a surge of flow in the injector to speed transfer of the sample. It may be used, depending upon the analysis, to sharpen peaks closer to the solvent's boiling point where cold trapping is ineffective and solvent effect is the main refocusing mechanism. Enter a value in the range of 5-1000 kPa (0.725-145 psi; 0.05-10 bar).
- Surge Duration This is the time for which the surge pressure is maintained. Enter a value in the range of 0.00-999.99 min. Typically, set to coincide with the **Splitless time**.

## **Septum Purge**

This field controls the septum purge for the injector. Septum purge is used to sweep the bottom of the septum to reduce contamination from the sample's analytes. This prevents carryover from run to run. Septum purge also prevents contamination of the injector from septum bleed.

- Purge Flow This field indicates the flow at which the septum is continuously flushed.
- **Constant Septum Purge** Select the check box to continuously flush the septum with a purge flow.
- Stop Purge Time This field is enabled if the Constant Septum Purge check box is cleared. You can then enter a time at which the septum purge ceases. The range is 0.00-999.99 min.

## **Carrier Mode**

In this field, you select the flow mode for the carrier gas.

The choice made here controls the parameters available in the Carrier Flow/ Carrier Pressure and Ramps field.

• **Constant Flow** — A single Flow field is available along with a check box to enable or disable it. The graph shows a horizontal line at the flow rate indicated. See Carrier Flow.

- **Constant Pressure** A single Pressure field is available along with a check box to enable or disable it. The graph shows a horizontal line at the pressure indicated. See Carrier Pressure.
- **Programmed Flow** The Flow program fields are displayed. The graph represents the ramps in the program. See Programmed Carrier Flow.
- **Programmed Pressure** The Pressure program fields are displayed. The graph represents the ramps in the program. See Programmed Carrier Pressure.

#### **Carrier Flow**

Use this field to set up flow when the **Constant flow** carrier mode has been selected.

Select the **Flow** check box to enable the Flow field. Enter a value in the range of 0.1-100 mL/min. If you clear the check box, the field is disabled and the graph becomes a horizontal line at a flow rate of zero.

#### **Carrier Pressure**

Use this field to set up pressure when the Constant pressure carrier mode has been selected.

Select the **Pressure** check box to enable the Pressure field. Enter a value in the range of 5-1000 kPa (0.725-145 psi; 0.05-10 bar). If you clear the check box, the field is disabled and the graph becomes a horizontal line at zero pressure.

#### **Programmed Carrier Flow**

Use this field to set up a flow program when **Programmed Flow** carrier mode has been selected.

In this mode you can program an initial constant flow rate field followed by up to three ramps.

Select the Flow enable check box to enable the flow.

Enable the number of flow ramps required by clicking on the 🕈 button.

Conversely, to reduce the number of rows on display, click on the 🔀 button as necessary.

The initial row is displayed by default and cannot be hidden.

If you wish to disable the ramps temporarily, clear the **Flow enable** check box. This disables the Flow column and the program is ignored as shown in the graph, which becomes a horizontal line at zero flow rate. The values entered are preserved and may be reactivated by selecting the check box once more.

The parameters in the timetable are:

- **Rate** Enter a value for the rate of flow rate change in the range of 0.001-100 mL/min<sup>2</sup>. This field is not available for the initial field of the program.
- **Flow** Enter a value for the constant flow rate field of the ramp in the range of 0.001-100 mL/min.
- **Hold Time** Enter a value for which the flow rate will be held. The range is 0.00-999.99 min.

#### **Programmed Carrier Pressure**

Use this field to set up a pressure program when **Programmed Pressure** carrier mode has been selected.

In this mode you can program an initial constant pressure rate field followed by up to three ramps.

Select the **Pressure enable** check box to enable the pressure.

Enable the number of pressure ramps required by clicking on the 🕈 button.

Conversely, to reduce the number of rows on display, click on the 🗡 button as necessary.

The initial row is displayed by default and cannot be hidden. If you wish to disable the Pressure ramps temporarily, clear the **Pressure enable** check box. This disables the Pressure column, and the program is ignored as shown in the graph, which becomes a horizontal line at zero pressure. The values entered are preserved and may be reactivated by selecting the check box once more.

The parameters in the timetable are:

- **Rate** Enter a value for the rate of pressure change in the range of 0.01-1000 kPa/min (0.001-145 psi; 0.0001-10 bar). This field is not available for the initial field of the program.
- **Pressure** Enter a value for the constant pressure field of the ramp in the range of 5-1000 kPa (0.725-145 psi; 0.05-10 bar).
- Hold Time Enter a time for which the pressure will be held. The range is 0.00-999.99 min.

## **Carrier Options**

This field includes the following parameters:

• Vacuum Compensation — Select the check box to enable the vacuum compensation feature. Use this only when the TRACE 1300 is connected to a mass spectrometer detector. When the check box is not selected, calculations are made for a normal GC detector, which is usually at atmospheric pressure.

• **Gas Saver** — Select the check box to enable the function in this field. Gas saver controls reduce carrier gas consumption, especially when a large split flow is used. It is set to come on at some point well after the injection to conserve gas.

Gas Saver parameters are:

- Gas Saver Flow Enter a value for the gas saver flow rate in the range of 5-500 mL/min.
- Gas Saver Time This is time into the run when the gas saver feature starts up. Enter a value in the range of 0.00-999.99 min.

#### **Helium Conservation**

This field includes the parameters for the helium conservation.

Helium conservation parameters are:

- Conservation enable Select the check box to enable the helium conservation feature.
- Helium delay Set the delay time before the helium conservation feature starts up. Enter a value in the range 0.00-999.99 min.

# **FID Page**

FID Page is the method editor for the Front/Back/Aux L/Aux R Flame Ionization Detector module.

This page includes the following fields:

#### Detector

Set the detector parameters in this field.

- Flame On Select the check box to ignite the flame. This can only happen if the Base Temperature is at least 150 °C and the Air and Hydrogen flows are enabled in the Flow field. If you clear this check box, the Air and Hydrogen check boxes are cleared automatically.
- Temperature Select the check box to enable the adjacent field. This controls the temperature of the detector. Enter a temperature in the range of 0-450 °C.
- **Ignition Threshold** If Flameout Retry is enabled, the flame will re-ignite if the signal current drops below this value. Enter a value in the range 0-10 pA.
- Flameout Retry Select the check box to enable this feature.

### Flow

These check boxes allow you to turn the flows on and off only.

- Air Select the check box to switch on the air flow and enable the adjacent flow rate field. Enter a value in the range of 5-500 ml/min.
- Hydrogen Select the check box to switch on the hydrogen flow and enable the adjacent flow rate field. Enter a value in the range of 1-100 mL/min.
- Make-up gas Select the check box to switch on the make-up flow and enable the adjacent flow rate field. Enter a value in the range of 1-50 mL/min.

#### Signal

- Acquire data Select this check box to enable the acquisition of the data.
- GC Peak width Specify the expected width of your GC peaks. For normal chromatography, select Standard GC peaks (> 1 s). For ultra-fast chromatography select Fast GC peaks (< 1 s). Because this parameter affects the data acquisition rate, selecting Fast for standard chromatography can result in noisier peaks.</li>

# **ECD** Page

ECD Page is the method editor for the Front/Back/Aux L/Aux R Electron Capture Detector module.

## Detector

Set the detector parameters in this field.

- Temperature Select the check box to enable the adjacent field. This controls the temperature of the detector. Enter a value in the range of 0-400 °C.
- Use Default Pulse Setting Select the check box to enable this feature. Reference current, Pulse amplitude, and Pulse width are set to optimum working values. When the check box is selected, the fields of these parameters are not enabled.
- **Reference Current** Enter a value for the cell reference current in the range of 0.1-1.5 nA.
- Pulse Amplitude Enter a value for the pulse amplitude in the range of 5-50 V.
- **Pulse Width** Select a value for the pulse width from the drop-down list. Enter a value in the range 0.1-2.0 μs.

Flow	
	The <b>Makeup</b> check box allows you to turn the flow on and off only.
	• Make-up gas — Select Makeup check box to enable the make-up gas flow and the adjacent field. The range for this entry depends on the gas being used. The range of the make-up gas flow rates is 5-500 mL/min for Nitrogen or Argon/Methane.
Signal	
	• Acquire data — Select this check box to enable the acquisition of the data.
	<ul> <li>GC Peak width — Specify the expected width of your GC peaks. For normal chromatography, select Standard GC peaks (&gt; 1 s). For ultra-fast chromatography select Fast GC peaks (&lt; 1 s). Because this parameter affects the data acquisition rate, selecting Fast for standard chromatography can result in noisier peaks.</li> </ul>
NPD Page	
	NPD Page is the method editor for the Front/Back/Aux L/Aux R Nitrogen Phosphorous Detector module.
	This page includes the following fields:
Detector	
	Set the detector parameters in this field.
	• Source Current — This is the current applied to heat the thermionic source. Enter a value in the range of 1.000-5.000 A.
	• Source Saver On — Check this box to enable the source saver function intended to reduce the consumption of the NPD source.
	• Temperature — Select the check box to enable the adjacent field. This controls the temperature of the detector. Enter a value in the range of 0-450 °C.
	• Use Default Polarization Voltage — Select the check box to enable this feature. When selected, the Polarization voltage field is not enabled.
	• <b>Polarization Voltage</b> — Enter a value in the range of 1-100 V.
Flow	
	These check boxes allow you to turn the gas flows on and off only.
	• Air — Select the check box to switch on the air flow and enable the adjacent flow rate field. Enter a value in the range of 5-500 mL/min.

- Hydrogen Select the check box to switch on the hydrogen flow and enable the adjacent flow rate field. Enter a value in the range of 0.1-10 mL/min.
- Make-up gas Select Makeup check box to switch on the make-up flow and enable the adjacent flow rate field. Enter a value in the range of 1.0-50.0 mL/min.

## Signal

- Acquire data Select this check box to enable the acquisition of the data.
- GC Peak width Specify the expected width of your GC peaks. For normal chromatography, select Standard GC peaks (> 1 s). For ultra-fast chromatography select Fast GC peaks (< 1 s). Because this parameter affects the data acquisition rate, selecting Fast for standard chromatography can result in noisier peaks.</li>

# **TCD Page**

TCD Page is the method editor for the Front/Back/Aux L/Aux R Thermal Conductivity Detector module.

This page includes the following fields:

### Detector

Set the detector parameters in this field.

- Temperature Select the check box to enable the adjacent field. This controls the temperature of the detector. Enter a value in the range of 0-400 °C.
   Typical temperature is 200 °C depending on the application used. Set the temperature to a higher value than the maximum temperature reached by the GC column oven during the analysis.
- Filament Power Select the check box to switch on the power to the filament.
- Filament Temperature Enter a value for the constant filament temperature in the range of 50-450 °C.

Filament temperatures should be kept 50-100 °C higher than the block temperature. The greater the difference the better the sensitivity. However, the usable difference between the block temperature and the filament temperature depends on the carrier gas used. The suggested  $\Delta T$  is:

- from 50 °C to 100 °C if the carrier gas is helium
- 100 °C if the carrier gas is nitrogen

#### Flow

The TCD Flow parameters are:

•	Make-up gas — Select Makeup check box to enable the make-up gas flow and the
	adjacent field. Enter a value for the make-up gas flow rate in the range of 0.5-5.0
	mL/min.

• **Carrier Source** — When two injectors are present, this parameter tells the chromatograph which inlet is connected to the TCD. It is used to protect the filaments on DGFC systems when the carrier gas supply is inadvertently shut off, for example following septum replacement. From the drop-down list, select among Back, Other, Aux 1-6.

### Signal

- Acquire data Select this check box to enable the acquisition of the data.
- GC Peak width Specify the expected width of your GC peaks. For normal chromatography, select Standard GC peaks (> 1 s). For ultra-fast chromatography select Fast GC peaks (< 1 s). Because this parameter affects the data acquisition rate, selecting Fast for standard chromatography can result in noisier peaks.</li>

# **FPD** Page

FPD Page is the method editor for the Front/Back/Aux L/Aux R Flame Photometric Detector module.

This page includes the following fields:

#### Detector

Set the detector parameters in this field.

- Flame On Select the check box to ignite the flame. This can only happen if the Base Temperature and Cell Temperature are at least 150 °C, and air and hydrogen flows are enabled in the Flow field. If you clear this check box, the Air and Hydrogen check boxes are cleared automatically.
- Base Temperature Select the check box to enable the adjacent field. This controls the temperature of the detector base. In the field enter a temperature in the range 0-450 °C.
- Cell Temperature Select the check box to enable the adjacent field. This controls the temperature of the detector. In the field enter a temperature in the range 0-200 °C.



**WARNING** The default Cell Temperature for the FPD is 150 °C. This is enough to prevent water generated by the flame to condensate inside the FPD body. Higher temperatures can be used but they may have adverse effects on PMT sensitivity.

	• <b>Ignition Threshold</b> — The flame will re-ignite if the signal current drops below this value. Set a value in the range 0.0-10.0 nA.
	• Use default PMT voltage — Select the check box if you want selecting the default PMT voltage of - 800 V. The PMT Voltage field is disabled.
	• <b>PMT Voltage</b> — This box is enabled when the Use default PMT voltage check box is clear. Set a value in the range from -800 V to -1200 V. This field is disabled when the Use default PMT voltage check box is selected.
Gas Flow	
	These check boxes allow you to turn the flows on and off only.
	• Air — Select the check box to switch on the air flow and enable the adjacent flow rate field. Enter a value in the range of 5-500 mL/min.
	• Hydrogen — Select the check box to switch on the hydrogen flow and enable the adjacent flow rate field. Enter a value in the range of 1-100 mL/min.
Signal	
	• Acquire data — Select this check box to enable the acquisition of the data.
	<ul> <li>GC Peak width — Specify the expected width of your GC peaks. For normal chromatography, select Standard GC peaks (&gt; 1 s). For ultra-fast chromatography select Fast GC peaks (&lt; 1 s). Because this parameter affects the data acquisition rate, selecting Fast for standard chromatography can result in noisier peaks.</li> </ul>
PDD Page	
	PDD Page is the method editor for the Front/Back/Aux L/Aux R Pulsed Discharge Detector module.
Detector	
	Set the detector parameters in this field.
	• <b>Pulse generator on</b> — Select the check box to turn on the pulse generator. This ignites the plasma discharge, which is done after stabilization and bakeout.
	• Temperature — Select the check box to enable the adjacent field. This controls the temperature of the detector. In the field enter a temperature in the range 0-450 °C.
Signal	

• Acquire data — Select this check box to enable the acquisition of the data.

GC Peak width — Specify the expected width of your GC peaks. For normal chromatography, select Standard GC peaks (> 1 s). For ultra-fast chromatography select Fast GC peaks (< 1 s). Because this parameter affects the data acquisition rate, selecting Fast for standard chromatography can result in noisier peaks.</li>

# **GDI Page**

GDI Page is the method editor for the Front/Back/Aux L/Aux R Generic Detector Interface for third-party detector.

**Note** On the right of Gas 1, Gas 2, and Gas 3, the type of gas (Air, Hydrogen, Nitrogen, Helium, Argon, or Argon/Methane) selected in the configuration page is indicated.

#### Detector

Set the detector parameter in this field.

• Temperature — Select the check box to enable the adjacent field. This controls the temperature of the detector. In the field enter a temperature in the range 0-450 °C.

#### **Gas Flow**

These check boxes allow you to turn the flows on and off only.

- Gas 1: Select the check box to switch on the flow of the detector gas connected to Gas 1 line, and enable the adjacent flow rate field. Set the value of the gas flow in mL/min according to the Full Scale value of the Gas 1 encapsulated flow restrictor.
- Gas 2: Select the check box to switch on the flow of the detector gas connected to Gas 2 line, and enable the adjacent flow rate field. Set the value of the gas flow in mL/min according to the Full Scale value of the Gas 2 encapsulated flow restrictor.
- Gas 3: Select the check box to switch on the flow of the detector gas connected to Gas 3 line, and enable the adjacent flow rate field. Set the value of the gas flow in mL/min according to the Full Scale value of the Gas 3encapsulated flow restrictor.

#### Signal

- Acquire data Select this check box to enable the acquisition of the data.
- GC Peak width Specify the expected width of your GC peaks. For normal chromatography, select Standard GC peaks (> 1 s). For ultra-fast chromatography select Fast GC peaks (< 1 s). Because this parameter affects the data acquisition rate, selecting Fast for standard chromatography can result in noisier peaks.</li>

# **Auxiliary Temperature Page**

Defines the temperature parameters of the external auxiliary temperature modules selected in the Instrument Configuration.

This page includes the following fields:

#### **Valve Oven Options**

Options of **Valve Oven** are: Valve Oven, HT Valve Oven, Aux Column Oven, Aux Heater 3, Aux Heater 4, and Methanizer.

- Valve Oven Controls the temperature of the main oven for working at low temperature. Enter a value up to 200 °C.
- **HT Valve Oven** Controls the temperature of the main oven for working at high temperature. Enter a value up to 300 °C.
- Aux Column Oven Controls the temperature of the secondary oven. Enter a value up to 250 °C.
- Aux Heater 3 Controls the temperature of the generic auxiliary heater 3. Enter a value up to 400 °C.
- Aux Heater 4 Controls the temperature of the generic auxiliary heater 4. Enter a value up to 400 °C.
- Methanizer Controls the temperature of the methanizer. Enter a value up to 400 °C.

#### **Auxiliary Temperature Control Options**

Options of **Auxiliary Temperature Control** are: Transfer Line 1, Transfer Line 2, Aux Heater 1, and Aux Heater 2.

- **Transfer Line 1** Controls the temperature of the MS transfer line. Enter a value up to 400 °C.
- **Transfer Line 2** Controls the temperature of a second MS transfer line. Enter a value up to 400 °C.
- Aux Heater 1 Controls the temperature of the generic auxiliary heater 1. Enter a value up to 400 °C.
- Aux Heater 2 Controls the temperature of the generic auxiliary heater 2. Enter a value up to 400 °C.

# **Auxiliary Carrier Page**

Defines the carrier gas parameters of the auxiliary Carrier Gas modules. Up to six auxiliary carrier lines from **Carrier 1** to **Carrier 6** can be selected.

Each carrier gas line includes the following field:

#### **Carrier Mode**

Defines the auxiliary carrier gas control mode to use: **Constant Flow**, **Constant Pressure**, **Programmed Flow**, and **Programmed Pressure**. Each mode activates or deactivates the dedicated parameters.

• **Constant Flow** — Use this field to set up flow when the **Constant flow** carrier mode has been selected.

Select the **Flow** check box to enable the Flow field. Enter a value in the range of 0.1-100 mL/min. If you clear the check box, the field is disabled and the graph becomes a horizontal line at a flow rate of zero.

• **Constant Pressure** — Use this field to set up pressure when the **Constant pressure** carrier mode has been selected.

Select the **Pressure** check box to enable the Pressure field. Enter a value in the range of 5-1000 kPa (0.725-145 psi; 0.05-10 bar). If you clear the check box, the field is disabled and the graph becomes a horizontal line at zero pressure.

- **Programmed Flow** Use this field to set up a flow program when **Programmed Flow** carrier mode has been selected.
  - d. Select the Flow enable check box to enable the flow.
  - e. Enable the number of flow ramps required by clicking the 🕈 button.
  - f. Conversely, to reduce the number of rows on display, click the 🔀 button as necessary.
  - g. The initial row is displayed by default and cannot be hidden.
  - h. If you wish to disable the ramps temporarily, clear the **Flow enable** check box. This disables the Flow column and the program is ignored as shown in the graph, which becomes a horizontal line at zero flow rate. The values entered are preserved and may be reactivated by selecting the check box once more.

The parameters in the timetable are as follows:

**Rate** — Enter a value for the rate of flow rate change in the range of  $0.001-100 \text{ mL/min}^2$ . This field is not available for the initial field of the program.

**Flow** — Enter a value for the constant flow rate field of the ramp in the range of 0.001-100 mL/min.

**Hold Time** — Enter a value for which the flow rate will be held. The range is 0.00-999.99 min.

• **Programmed Pressure** — Use this field to set up a flow program when **Programmed Pressure carrier** mode has been selected.

In this mode you can program an initial constant pressure rate field followed by up to three ramps.

- a. Select the **Pressure enable** check box to enable the pressure.
- b. Enable the number of pressure ramps required by clicking on the 🔂 button.
- c. Conversely, to reduce the number of rows on display, click on the ⊠ button as necessary.
- d. The initial row is displayed by default and cannot be hidden. If you wish to disable the Pressure ramps temporarily, clear the **Pressure enable** check box. This disables the Pressure column, and the program is ignored as shown in the graph, which becomes a horizontal line at zero pressure. The values entered are preserved and may be reactivated by selecting the check box once more.

The parameters in the timetable are as follows.

**Rate** — Enter a value for the rate of pressure change in the range of 0.01-1000 kPa/min (0.001-145 psi; 0.0001-10 bar). This field is not available for the initial field of the program.

**Pressure** — Enter a value for the constant pressure field of the ramp in the range of 5-1000 kPa (0.725-145 psi; 0.05-10 bar).

**Hold Time** — Enter a time for which the pressure will be held. The range is 0.00-999.99 min.

## **Run Table Page**

You can use this page to set up detector, valve, and external events to occur at the Prep-run or during a run. Events displayed on the Run Table page are from selections made in the Run-Time Event screen.

This page includes the following fields:

#### **Initial Values**

In this field you can activate the externally controlled devices set in External Event field. See also "Run-Time Event" on page 131 for details.

Select the option button to activate the associated fields.

#### **Event Program Timetable**

Events are sorted into time order automatically. The table has three columns: **Time**, **Item** and **Setting**.

- Time This shows the time in the run at which the event will take place.
- Item The Item column shows the event type and identifies the external event or valve number.
- Action This column indicates Action associated with the event, for example switching a lamp on or off.

#### **Adding Events**

Click **Add** button to view the Run-Time Event dialog window. Run items you select from this dialog are added to the Event Program Timetable.

#### **Modifying Events**

Click Modify button to edit a selected run item listed in the Event Program Timetable.

#### **Removing Events**

Select an item in the Event Program Timetable. Click **Remove** button to remove the selected run item from the run table.

# **Run-Time Event**

The Run-Time Event window is displayed when the Add button is selected.

There are four types of events: Detector, Valve Oven, External, and Aux Carrier.

#### **Event Time**

Select either the **Prep Run** or **Run Time** option button. When you select the **Prep Run** option button, the time field is disabled.

Select the **Run Time** option button to enable the adjacent field. Enter the time at which the event is to occur in the range of 0.00-999.99 min.

#### Event

Select the event type among Detector, Valve Oven, External, and Aux Carrier.

#### **Detector Event**

Select **Detector** to activate the associated fields. In the Detector field, choose the detector to which the event is to be applied. This selection enables the action appropriate to the type of detector.

The actions are:

- Autozero Applies to all detector types. Select this option to zero the detector output at the given time.
- Neg. Polarity Applies to TCD only. The entry will invert the polarity at the given time. Choose On or Off.
- Source Current Applies to NPD only. The entry will change the source current at the given time. Choose On or Off.

#### Valve Oven Event

Select **Valve Oven** to activate the associated fields. In the Valve Oven field, set the condition of the desired valve controlled into the TRACE 1310 Auxiliary Oven (Valve Oven).

The options are:

- Number Selects the number of the valve in the range of 1-8.
- Valve type Selects Sampling or Switching according to the valve type.
- Setting Sets the event to On or Off.

#### **External Event**

Select **External** to activate the associated fields. In the External field, set the default condition for up to eight externally controlled devices.

The options are:

- Number Select the number of the external event in the range of 1-8.
- Setting Set the event to On or Off.

**Note** When the chromatograph is in Standby mode, each device will return to the default condition set here. The option selected is visualized and enabled in Initial Values field.

#### **Aux Carrier Event**

Select **Aux Carrier** to activate the associated fields. In the Aux Carrier field, set the default condition for up to six auxiliary carrier lines.

The options are:

- Number Selects the number of the auxiliary carrier line in the range of 1-6.
- Carrier off Check this radio button to turn off the auxiliary carrier of the selected line.
- **Pressure** Check this radio button to set the desired pressure for the auxiliary carrier of the selected line.
- Flow Check this radio button to set the desired flow for the auxiliary carrier of the selected line.

# **Oven**

This chapter provides the instruction for programming the oven parameters.

#### Contents

- Oven Capabilities
- Using Oven Parameters
- Setting Up a Single Ramp Temperature Program
- Setting Up a Multiple Ramp Temperature Program

## **Oven Capabilities**

The oven has the following capabilities:

- Maximum temperature of 450 °C.
- Maximum temperature increase rate of 125 °C/min.
- 32 linear temperature ramps and 33 plateaus.
- Minimum operating temperature of a few degrees above ambient, which is obtained by a couple of modulated cooling flaps controlled by the GC.
- Temperature control through heater control, fine control of hot air exhaust, and ambient air intake.
- Allows the separation of moderately volatile components on thick film capillary columns at near ambient temperatures without the use of a cryogenic system.
- With a cryogenic option installed, the oven temperatures can reach –50 °C with carbon dioxide, and –100 °C with liquid nitrogen.

# **Using Oven Parameters**

This section details the parameters for programming the oven temperature, from an initial temperature to a final temperature, using up to 32 ramps during the analytical run.

It is possible to set a single (isothermal) or multiple ramp programs.

**Temperature** — Defines the initial temperature. The range is On/Off; 0–450 °C. With a cryogenic system, the ranges are from –100 to 450 °C with liquid nitrogen, and from –50 to 450 °C with carbon dioxide. Select **On** to display the actual and setpoint values.

**Initial time** — Defines the time the oven remains at the starting temperature after a programmed run has begun. Enter a value in the range of 0-999.99 min.

**Ramp 1** — Defines the temperature ramp rate in °C/min to reach the final temperature. The range is On/Off; 0.0–125 °C/min. Select **On** to enable a temperature ramp.

**Final temperature 1** — Defines the temperature the column oven will reach at the end of the heating or cooling ramp. This parameter is visualized only if **Ramp 1** is **On**. Enter a value in the range of 0-450 °C. With a cryogenic system, the ranges are from -100 to 450 °C with liquid nitrogen, and from -50 to 450 °C with carbon dioxide.

**Final time 1** — Defines how long (in minutes) the oven will maintain the final temperature of the ramp. Enter a value in the range 0.00–999.99 min.

**Ramp 2 to 32** — After you program the first ramp, the menu adds the **Ramp 2** parameter lines. The range is 0.0–120 °C/min. If you do not want an additional ramp, leave this parameter set to **Off**. To program the ramp, select **On**. **Final temp** and **Final time** parameters for the ramp will be added. You can repeat this process to program up to 32 temperature ramps.

**Final temperature 2 to 33** — Defines the temperature the column oven will reach at the end of the relevant ramp. Enter a value in the range of 0-450 °C. With a cryogenic system, the ranges are from -100 to 450 °C with liquid nitrogen, and from -50 to 450 °C with carbon dioxide.

**Final time 2 to 33** — Defines how long (in minutes) the oven will maintain the final temperature of the ramp. Enter a value in the range of 0.00–999.99 min.

**Post run temp** — Defines the temperature the oven will reach after the end of the analytical run. The range is On/Off; 0–450 °C. With a cryogenic system, the ranges are from –100 to 450 °C with liquid nitrogen, and from –50 to 450 °C with carbon dioxide. Select **Off** if you do not want a post run temperature. Select **On** to display the setpoint value and the parameters **Post run temp**, **Post run time**, and **Post pressure**.

**Post run time** — Defines the time the oven maintains the post run temperature. Enter a value in the range of 0.00–999.99 min.

**Post pressure** — Defines the pressure for the front or back carrier during the Post run time when the system operates in **Constant pressure** or **Programmed pressure** mode. Enter a value in the range 2.5–1000 kPa.

### Setting Up a Single Ramp Temperature Program

This program raises the initial oven temperature to a specified final temperature at a specified rate and maintains the final temperature for a specified time.

#### To setup a single ramp temperature program

- 1. Select **Oven** to open the oven menu.
- 2. Enter the Initial temperature.
- 3. Enter the **Initial time** that the oven will maintain the initial temperature.
- 4. Select and set **Ramp 1** to **On**. Enter the ramp rate in °C/minute for the oven to reach the ramp's Final temp.
- 5. Enter the Final temp 1 for the ramp.
- 6. Specify in **Final time 1** how long the oven will maintain the Final temperature.
- 7. To end the single ramp program, set Ramp 2 to Off.

#### Setting Up a Multiple Ramp Temperature Program

#### To setup a multiple ramp temperature program

This program raises the initial oven temperature to a specified final temperature through up to 32 ramps, each having a specified ramp rate, time, and temperature.

- 1. Select **Oven** to open the Oven menu.
- 2. Enter the Initial temperature.
- 3. Enter the **Initial time** that the oven will maintain the initial temperature.
- 4. Select and set **Ramp 1** to **On**. Enter the ramp rate in °C/minute for the oven to reach the ramp's Final temp.
- 5. Enter the **Final temp 1** for the ramp.
- 6. Specify in **Final time 1** how long the oven will maintain the Final temperature.
- 7. Select and set **Ramp 2** to **On**. Enter the ramp rate for the second temperature ramp.
- 8. Enter the **Final temp 2** for the second ramp.
- 9. Specify in Final time 2 how long the oven will maintain the Final temp.
- 10. To end the multiple ramp temperature program, leave **Ramp 3** set to **Off**. To add additional oven ramps, repeat the steps 7 through **9**.

# **Split/Splitless Injector (SSL) Module**

This chapter describes the Split/Splitless Injector (SSL) module and provides instructions for programming the injector parameters.

#### Contents

- Module Overview
- SSL Injection Technique
- Large Volume Splitless Injector (LVSL)
- Consumables
- Using SSL Parameters

## **Module Overview**

The split/splitless injector module includes the injector body, heater body, split and purge vent valves, filters, digital pneumatics for the control of the carrier gas, and fittings for the connection to the analytical column.

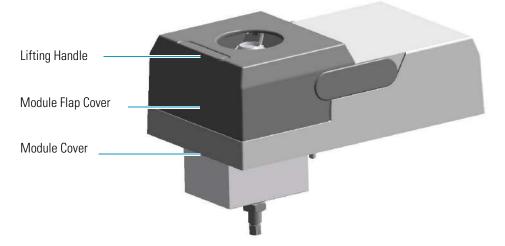


Figure 88. Split/Splitless Injector Module

The module and injector components are shown in Figure 89 and Figure 90.

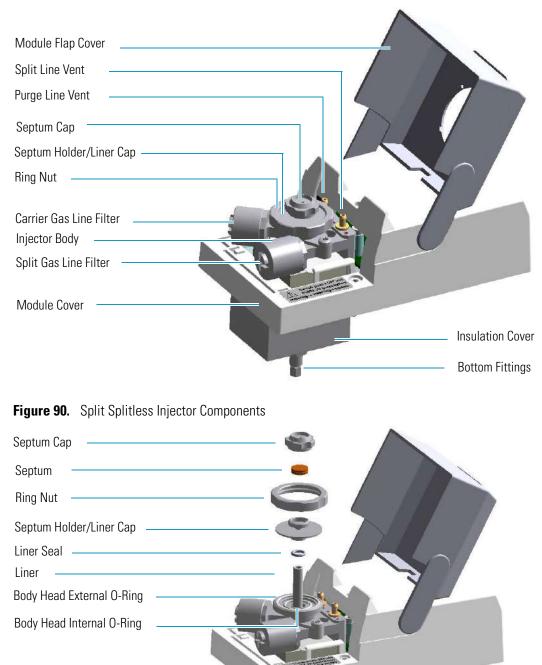


Figure 89. Split Splitless Injector Module Components



Base Seal \_\_\_\_\_ Washer \_\_\_\_\_

Retaining Nut

Terminal Fitting for Capillary Column \_

**Bottom Fittings** 

# **SSL** Injection Technique

The split/splitless (SSL) injector is used for split, splitless, or surged splitless injections. Heavy component discrimination is minimized with optimized sample transfer to the column. You can use capillary and wide-bore columns. You can choose the operating mode from the injector parameter list.

## **Split Mode**

Split injection mode is suitable for high-concentration sample analysis, headspace analysis, and isothermal analysis. Peak shapes in the chromatogram are generally very sharp due to the rapid sample transfer into the column. You inject the sample into a glass liner inside the heated vaporization chamber. In the chamber, the sample undergoes rapid evaporation. The relatively high gas flow through the injector carries the vaporized sample rapidly down toward the head of the column.

Only a part of the sample transfers into the column. The rest discharges through the split line. The ratio of the split flow to the column flow (the **split ratio**) determines the amount of sample that enters the chromatographic column.

The septum is continuously flushed throughout the analysis, which reduces contamination from the sample's analytes.

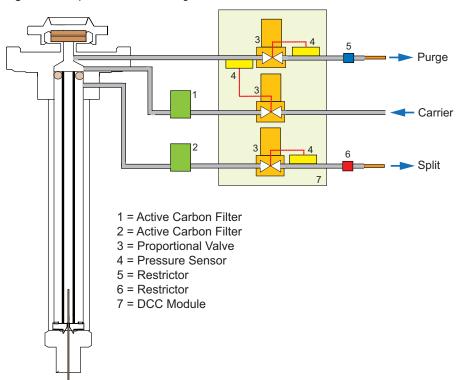


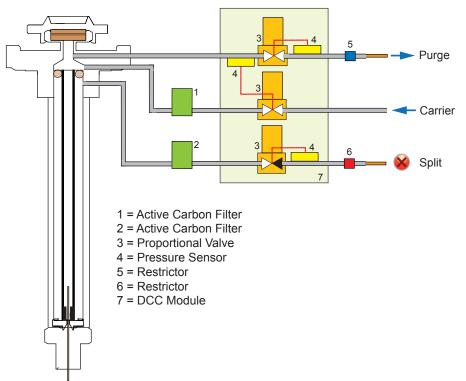
Figure 91. Split Mode Block Diagram

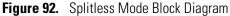
## **Splitless Mode**

Splitless injection mode is suitable for the analysis of compounds present in low concentrations.

The splitless injection allows the entire sample to enter the column. The split line is closed during the sample injection and transfer to the column. Once the transfer is over, the split line reopens to flush the vaporization chamber of any remaining sample vapors. The time required to transfer the vaporized sample from the injector to the column is the **splitless time**. At the end of the splitless period, the split valve reopens and the split flow flushes the injector of any remaining sample vapors. The absolute split flow is not important. It need only be sufficient to purge the injector.

A purge flow can continuously flush the septum throughout the analysis, which reduces contamination from the sample's analytes.





#### **Surged Splitless Mode**

In this mode, a carrier gas pressure surge activates during the injection phase for a preset time. The pressure applied during the splitless time produces a surge of the flow in the injector, and accelerates the transfer process of the sample substances from the injector to the column. This avoids the broadening of the sample band, and the risk of the injector overloading. A purge flow can continuously flush the septum throughout the analysis, which reduces contamination from sample's analytes.

# Large Volume Splitless Injector (LVSL)

The LV Splitless injector is a setup of the standard splitless injector, where the introduction of large amount of liquid samples can be performed manually, or with the TriPlus RSH, TriPlus 100 Liquid Sampler, or AI/AS 1310 autosampler.

# **LV Splitless Injection Requirements**

Large Volume-Splitless injection requires the SSL-LV Kit for TRACE 1300/TRACE 1310 (PN 19050725).

The kit incudes the following parts:

- Two dedicated bypass for the filters and relative O-rings
- A splitless liner
- A deactivated press-fit connector
- An uncoated pre-column (5 m x 0.32 mm i.d.) with a capacity for retaining an amount of liquid at least corresponding to the volume of sample injected

**Note** To correctly perform the Large Volume Splitless injection be sure to replace the filters on the carrier and split lines with the provided bypass included into the SSL-LV Kit.

# **The Large Volume Splitless Injection Technique**

In the standard splitless mode the injector can accept and quantitatively transfer to the column sample volumes of up to 5 mL. Splitless injections of higher amounts are possible by the Concurrent Solvent Recondensation technique (CSR LVSL).

The LVSL technique can be summarized by five key steps.

- 1. Injection with liquid band formation.
- 2. Auto pressure surge.
- 3. Recondensation of the solvent vapors in the pre-column concurrent with solvent evaporation in the injector.
- 4. Solutes transfer.
- 5. Solvent evaporation in the pre-column.

The mechanism of the concurrent solvent recondensation is shown in Figure 93.

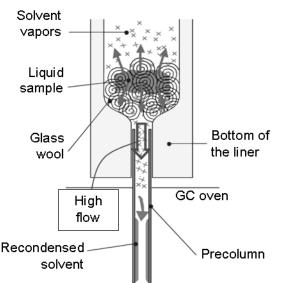


Figure 93. LVSL Mechanism of Concurrent Solvent Recondensation

The injection is performed through a cool syringe needle combining a short needle penetration in the injector with a cool injector head. In this way the sample liquid leaves the needle as a band and moves at high velocity reaching the bottom of the liner without substantial evaporation.

When the large amount of liquid is injected and collected on the bottom of the liner (packing material or laminar cup) a violent evaporation starts, generating a large volume of vapors. Solvent evaporation strongly increases pressure in the injector driving the first vapors into the pre-column (auto pressure surge).



**CAUTION** The auto pressure surge presupposes the closure of the septum purge outlet during the splitless period in order to prevent escape of carrier gas and of solvent vapor.

Since the pre-column is kept at a temperature below the boiling point of the solvent, vapors quickly recondensed. The elevated pressure drop between the high-pressure liner and the low-pressure recondensation site (solvent vapor pressure) produces a high flow from the hot injector into the cold pre-column.

After the solvent evaporation in the hot liner is completed, also high-boiling components are transferred to the column. In conventional splitless this happens during the splitless period.

Almost all the solvent is evaporated in the injector and recondensed in the pre-column. The solvent must now evaporate again in the pre-column. To keep the evaporation under control the oven temperature must be maintained at its initial temperature until the evaporation is completed.

Volatile solutes are reconcentrated by solvent trapping while high-boiling components are refocused by the retention gap effect. As the material is spread in a zone of a retentive power far below that of the separation column, bands are focused at the entrance of the coated separation column.

# Consumables

The consumables required for this injector are the septum, the glass liner, and the o-rings.

# Septum

You should always use good quality septa, such as the BTO septum supplied with the TRACE 1300/TRACE 1310. Such septa resist deformation, have longer life expectancy, and have a low bleed level, even at high temperatures.

SSL injector is compatible with the **Merlin Microseal™ High Pressure Valve** instead of the standard septum.

# Liner

The sample injection mode used determines the choice of liner to install into the injector body. An appropriate liner must ensure complete sample vaporization and contain the entire volume of the vaporized sample without reacting with it.

Thermo Scientific Chromatography Data Systems include the **Vapor Volume Calculator** that rapidly calculates the expansion volume of several factors (solvent, injected liquid volume, temperature, and inlet pressure) to help you determine if a liner dimension is suitable for a method.

## **Split Liners**

Select an appropriate liner from Table 5.

 Table 5.
 Split Liners

No	Liner	Description
1		Deactivated liner 4-mm ID; Glass Wool; 900 µL theoretical volume.
2		Deactivated empty liner; 4-mm ID.
3		Mini Lam Deactivated empty liner; 4-mm ID.

#### **Splitless Liners**

Select an appropriate liner from Table 6.

Table 6. Splitless Liners

No	Liner	Description
1		Deactivated Single Tapered Glass Wool; 900 µL theoretical volume.
2	-	Deactivated Single Tapered empty liner.

#### **HS/SPME** Liners

Select an appropriate liner from Table 7.

**Table 7.**HS/SPME Liners

No	Liner	Description
1		Deactivated empty liner; 1.2-mm ID.

# **O-rings**

The internal (carrier line) and external (purge line) o-rings of the body head must be replaced if there are leaks. To replace the o-rings, please refer to *TRACE 1300/TRACE 1310 Hardware Manual*.

# **Using SSL Parameters**

This menu includes the operating parameters for the split/splitless module. Editable parameters vary with the operating mode: split, splitless, surged splitless, and the flow mode: constant flow, constant pressure, programmed flow, programmed pressure.

- Carrier Gas Parameters
- Injection Mode
- Inlet Parameters
- Purge Parameters
- Surge Parameters
- Setting Gas Parameters
- Setting Parameters for the Split Mode
- Setting Parameters for the Splitless Mode
- Setting Parameters for the Surged Splitless Mode

The following sections list and describe the parameters controlling the front/back SSL injector. They are:

# **Carrier Gas Parameters**

Set the carrier gas control parameters. Visualized parameters change according to the **Flow Mode** set.

**Pressure** — Defines the actual and setpoint pressure of the carrier gas. The range is On/Off; 5-1000 kPa (0.725-145 psi; 0.05-10 bar). This line is not editable when the Constant Flow or Programmed Flow mode is selected.

**Column Flow** — Defines the carrier gas flow rate through the column. The range is On/Off; 0.01-100 mL/min. Select **On** to display the actual and setpoint values. Select **Off** or **0** to turn off all inlet flows. This line is not editable when the Constant Pressure or Programmed Pressure mode is selected.

**Flow Mode** — Defines the carrier gas control mode to use. Each mode activates or deactivates the dedicated parameters.

- **Constant Flow** The column flow is kept constant throughout the analysis. The pressure at the column head will change with the column temperature to maintain a consistent flow.
- **Constant Pressure** The pressure at the column head is kept constant throughout the analysis. During a temperature program, the column flow decreases due to the increase of the carrier gas viscosity.
- **Programmed Flow** The column flow rate can be programmed to change during the analytical run for up to three flow ramps.

The parameters are:

- Initial Flow Defines the beginning flow rate.
- Initial Time Defines how long the Initial Flow is maintained.
- Ramp 1 The ramp rate in mL/min<sup>2</sup> to reach the final flow rate. Select On to enable the ramp and display the setpoint value.
- Final Flow The final flow rate the carrier gas will reach at the end of the ramp rate.
- **Final Time** Defines how long the corresponding Final flow must be maintained.
- Ramp 2-3 To program additional ramps, select On and enter the ramp rates in mL/min<sup>2</sup>. The Final Flow and Final Time menu items for the ramp are displayed. The ranges and functions of these menu items are identical to the Final Flow and Final Time menu items for Ramp 1.
- **Programmed Pressure** The inlet pressure can be programmed to change during the analytical run up to three pressure ramps.

The parameters are:

- Initial Pressure Defines the initial pressure.
- Initial Time Defines how long the Initial Pressure is maintained.
- Ramp 1 Defines the ramp pressure in kPa/min to reach the Final Pressure. Select
   On to enable the ramp and display the setpoint value.

- **Final Pressure** Defines the final pressure the carrier gas will reach at the end of the ramp rate.
- Final Time Defines how long the corresponding final pressure must be maintained.
- Ramp 2-3 To program additional ramps, select On and enter the ramp rates in kPa/min. The Final Pressure and Final Time menu items for the ramp are displayed. The ranges and functions of these menu items are identical to the Final Pressure and Final Time menu items for Ramp 1.

**Linear Velocity** — The calculated velocity of the carrier gas through the column, expressed in cm/s. It is not editable.

**Void Time** — The elution time of an un-retained peak, expressed in seconds. It is not editable.

**Gas Saver** — This function reduces carrier gas consumption. The range is On/Off; 5–500 mL/min. Select **On** to turn on the gas saver flow and display the setpoint values. Select **Off** to turn off the gas saver flow. The flow is retained in memory.

**Gas Saver Time** — Defines the time in the run when the gas saver function starts to operate. Usually it starts after the injection to conserve gas. Set a value from 0.00 to 999.99 min. This line does not appear if Gas saver flow is Off.

**Vacuum Comp.** — Use this parameter only when the TRACE 1300/TRACE 1310 is coupled with a mass detector to compensate for vacuum column outlet. The range is On/Off.

# **Injection Mode**

Choose which injection mode to use with the SSL injector. Each mode activates or deactivates the dedicated parameters.

**Split** — The carrier flow is split in the injection port with the bulk going out the split vent. The split vent remains open all the time.

**Splitless** — Closes the split vent during injection to drive the entire sample into the column. Splitless times of about 1 minute are typical.

**Surged Splitless** — Same as **Splitless** but can also program a surge during an injection. Surge starts at Prep Run and continues until the surge duration time is finished. Surge is further defined in next group of controls.

## **Inlet Parameters**

The SSL injection parameters are described below:

**Temperature** — Defines the setpoint for the injector's temperature. Depending on the injection mode you select, set a temperature high enough to vaporize the sample and the solvent. Enter a value for the inlet temperature in the range of 0-400 °C.

**Split Flow** — Enter a value in the range of 5-1250 mL/min. The **Split Ratio** is adjusted automatically. In addition, this value is governed by the initial column flow rate entered on the associated carrier gas control. If the flow rate is changed, the Split Flow value is adjusted so that the Split Ratio is maintained. However, if the Split Flow value then falls outside its limits, a warning is generated.

**Split Ratio** — Enabled when the injection mode is set to **Split**. It is also associated to the Flow Mode set to either Constant Flow or Programmed Flow. Specify the ratio of split flow to column flow. Calculate the split ratio: Split Ratio = (split flow) / (column flow). Enter a value in the range of 1-12500. The Split Flow entry is adjusted automatically.

**Splitless Time** — Enabled when the injection mode is set to either **Splitless** or **Surged Splitless**. Specify the length of time the split valve remains closed after a splitless injection. Enter a value in the range of 0.00-999.99 min. The timer begins at the start of the run. The split vent reopens when the splitless time ends.

## **Purge Parameters**

Purge parameters are available when the injection mode is set to either **Splitless** or **Surged Splitless**.

**Constant Septum Purge** — Controls the septum purge for the injector. The range is On; Off. Select **On** to activate the function to continuously flush the septum with a purge flow.

**Note** The purge flow, in the range of 0.5-50 mL/min, must be set in the Configuration Page of the injector module.

**Stop Purge For** — Enabled if the Constant Septum Purge parameter is set to On. You can then enter a time from 0.00 to 999.99 min at which the septum purge ceases.

# **Surge Parameters**

Surge parameters are enabled when the injection mode is set to Surged Splitless.

**Surge Pressure** — Defines the pressure applied during the splitless time to produce a surge of flow in the injector to speed the transfer of the sample. Enter a value in the range of 5-1000 kPa (0.725-145 psi; 0.05-10 bar).

**Surge Duration** — Defines the time that the surge pressure is maintained. Enter a value in the range of 0.00-999.99 min. Typically, set to coincide with the **Splitless time**.

# **Setting Gas Parameters**

Before starting, check that the carrier gas type is correct for the analysis.

#### ✤ To set-up gas parameters

- 1. Program the carrier gas flow.
  - a. Select Flow Mode.
  - b. Choose the mode you want choosing among Constant flow, Constant Pressure, Programmed Flow or Programmed Pressure.
  - c. Enter the initial Flow, or Pressure.
    - i. If you select the **Constant Flow** mode, enter the desired **Column Flow** value. The necessary pressure is calculated and adjusted to maintain the constant flow.
    - ii. If you select **Constant Pressure** mode, enter the desired **Pressure** value.
  - d. Enter a **Programmed Flow/Programmed Pressure**.

**Note** When you select Programmed Flow or Programmed Pressure mode, the menu for the carrier contains parameters for up to three program ramps.

- i. Select **Progr Flow/Pressure**, scroll to Initial Flow/Press and enter the desired value. Press Enter.
- ii. Scroll to Initial Time and enter a value. This parameter ends the initial part of the program.
- e. Program the Ramps.
  - i. Scroll to Ramp 1 and enter the value.
  - ii. Scroll to Final Flow 1/Pressure 1 and enter the final value for the ramp.
  - iii. Scroll to Final Time 1 and enter the final time for Ramp 1. This operation ends the first ramp setting.
  - iv. If you do not want a second ramp, leave Ramp 2 set to **Off**. To enter a second ramp, scroll to Ramp 2 and enter the value.
  - v. Scroll to Final Flow 2/Pressure 2 and enter the final value for the ramp.
  - vi. Scroll to Final Time 2 and enter the final time for Ramp 2. This operation ends the second ramp setting.
  - vii. If you do not want a third ramp, leave Ramp 3 set to **Off**. To enter a third ramp, scroll to Ramp 3 and enter the value.
  - viii. Scroll to Final Flow 3/Pressure 3 and enter the final value for the ramp.
  - ix. Scroll to Final Time 3 and enter the final time for Ramp 3. This operation ends the third ramp setting.

2. If the TRACE 1300/TRACE 1310 is working with a mass detector, set **Vacuum Compensation** to **On** to compensate for vacuum column outlet.

# **Setting Parameters for the Split Mode**

Before starting, verify that the correct liner is installed into the injector body and the system is free of leak.

#### \* To setup a Split injection

- 1. Program the carrier gas flow.
- 2. From the mode list, choose **Split**.
- 3. Set the injector **Temperature**.
- 4. If carrier **Flow** mode (Programmed or Constant) is selected, specify the **Split Flow** or **Split Ratio**.
  - a. If you want a specific **Split Flow**, enter that value. **Split Ratio** will be calculated.
  - b. If you want a specific Split Ratio, enter that value. Split Flow will be calculated.
- 5. If desired, turn on Gas Saver and set Gas Saver Time after the injection time.

## **Setting Parameters for the Splitless Mode**

Before starting, verify that the correct liner is installed into the injector body and the system is free of leaks.

- To setup a Splitless injection
- 1. Program the carrier gas flow.
- 2. From the mode list, choose **Splitless**.
- 3. Set the injector Temperature.
- 4. Enter the **Splitless Time**.
- 5. If desired turn on **Constant Septum Purge** and enter into **Stop Purge For** how many minutes elapse before restarting the purge.
- 6. If desired, turn on Gas Saver and set Gas Saver Time after the injection time.

# **Setting Parameters for the Surged Splitless Mode**

Before starting, verify that the correct liner is installed into the injector body and the system is free of leaks.

#### ✤ To set up a Surged Splitless injection

- 1. Program the carrier gas flow.
- 2. From the Mode list, choose Surged Splitless.
- 3. Set the injector **Temperature**.
- 4. Enter the **Splitless Time**.
- 5. If desired turn on **Constant Septum Purge** and enter into **Stop Purge For** how many minutes elapse before restarting the purge.
- 6. Set the values for Surge Pressure and Surge Duration.
- 7. If desired, turn on Gas Saver and set Gas Saver Time after the injection time.

7

# **Split/Splitless Backflush Injector (SSLBKF) Module**

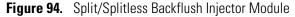
This chapter describes the Split/Splitless Injector module for **backflush** applications (SSLBKF) and provides instructions to program the injector parameters.

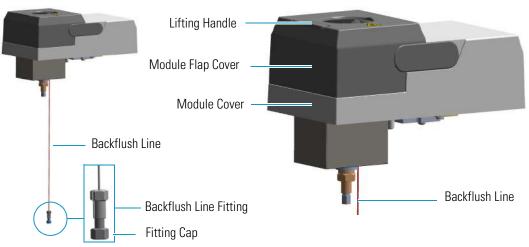
#### Contents

- Module Overview
- Backflush Mode
- Consumables
- Using SSLBKF Parameters
- Connecting the SSL Backflush System

# **Module Overview**

The SSL Backflush module includes the injector body, heater body, split and purge vent valves, filters, digital pneumatics for the control of the carrier gas, backflush control, backflush line, and fittings for the connection to the analytical column.





The module and injector components are shown in Figure 95 and Figure 96.

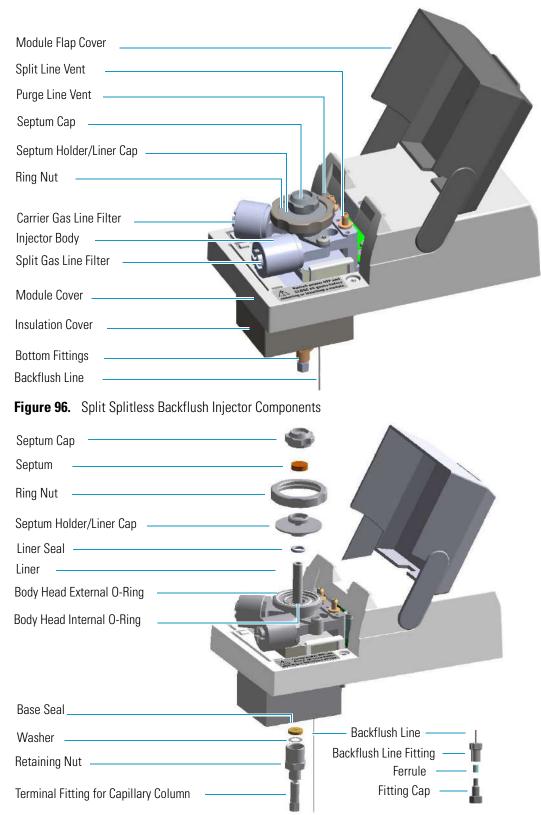


Figure 95. Split Splitless Backflush Injector Module Components

# **Backflush Mode**

This operating mode allows elimination of the solvent before the sample enters the column and the heavy part of the sample, which is not relevant for the analysis.

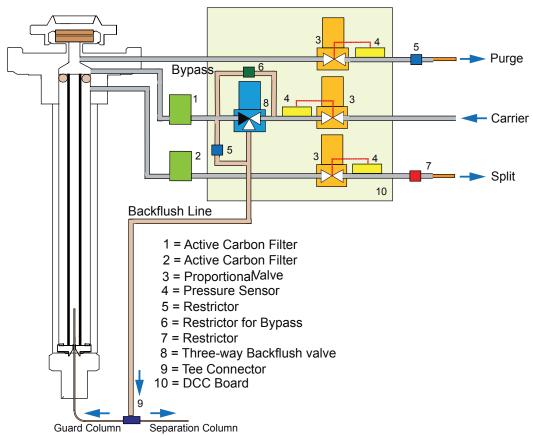


Figure 97. SSL Backflush Mode Block Diagram

# **Split Mode**

In this mode the sample is injected into the liner in hot condition by keeping the injection temperature suitable to volatilies the compounds.

During split injection the split and purge valves remain open throughout the run. Only a part of the sample transfers into the column. The rest discharges through the split line. The ratio of the split flow to the column flow (the **split ratio**) determines the amount of sample that enters the chromatographic column.

# **Splitless Mode**

In this mode the sample is injected into the liner in hot condition by keeping the injection temperature suitable to volatilize the compounds.

The splitless injection allows the entire sample to enter the column. The split line is closed during the sample injection and transfer to the column. Once the transfer is over, the split line reopens to flush the vaporization chamber of any remaining sample vapors. The time required to transfer the vaporized sample from the injector to the column is the **splitless time**. At the end of the splitless period, the split valve reopens and the split flow flushes the injector of any remaining sample vapors.

# **Surged Splitless Mode**

In this mode the sample is injected into the liner in hot condition by keeping the injection temperature suitable to volatilize the compounds.

A carrier gas pressure surge activates during the injection phase for a preset time. The pressure applied during the splitless time produces a surge of flow in the injector, and accelerates the transfer process of the sample substances from the injector to the column. This avoids the broadening of the sample band and the risk of the injector overloading.

A constant septum purge flow can continuously flush the septum throughout the analysis, which reduces contamination from sample's analytes.

# Consumables

The consumables required for this injector are the septum, the glass liner, and the o-rings.

# Septum

You should always use good quality septa, such as the BTO septum supplied with the TRACE 1300/TRACE 1310. Such septa resist deformation, have longer life expectancy, and have a low bleed level, even at high temperatures.

SSL injector is compatible with the **Merlin Microseal™ High Pressure Valve** instead of the standard septum.

# Liner

The sample injection mode used determines the choice of liner to install into the injector body. An appropriate liner must ensure complete sample vaporization and contain the entire volume of the vaporized sample without reacting with it. Thermo Scientific Chromatography Data Systems include the **Vapor Volume Calculator** that rapidly calculates the expansion volume of several factors (solvent, injected liquid volume, temperature, and inlet pressure) to help you determine if a liner dimension is suitable for a method.

#### **Split Liners**

Select an appropriate liner from Table 8.

Table 8. Split Liners

No	Liner	Description
1		Deactivated liner; 4-mm ID; Glass Wool; 900 µL theoretical volume.
2		Deactivated empty liner; 4-mm ID.
3		Mini Lam Deactivated empty liner 4-mm ID.

#### **Splitless Liners**

Select an appropriate liner from Table 9.

 Table 9.
 Splitless Liners

No	Liner	Description
1		Deactivated Single Tapered Glass Wool; 900 µL theoretical volume.
2	-(	Deactivated Single Tapered empty liner.

#### **HS/SPME** Liners

Select an appropriate liner from Table 10.

Table 10. HS/SPME Liners

No	Liner	Description
1	4	Deactivated empty liner; 1.2-mm ID.

# **O-rings**

The internal (carrier line) and external (purge line) o-rings of the body head must be replaced if there are leaks. To replace the o-rings, please refer to *TRACE 1300/TRACE 1310 Hardware Manual*.

# **Using SSLBKF Parameters**

This menu includes the operating parameters for the split/splitless module for backflush applications. Editable parameters vary with the operating mode: split, splitless, surged splitless, and the flow mode: constant flow, constant pressure, programmed flow, programmed pressure.

- Carrier Gas Parameters
- Injection Mode
- Inlet Parameters
- Purge Parameters
- Surge Parameters
- Setting Gas Parameters
- Setting Parameters for Backflush
- Setting Parameters for the Split Mode
- Setting Parameters for the Splitless Mode
- Setting Parameters for the Surged Splitless Mode

The following sections list and describe the parameters controlling the front/back SSLBKF injector. They are:

## **Carrier Gas Parameters**

Set the carrier gas control parameters. Visualized parameters change according to the **Flow Mode** set.

**Pressure** — Defines the actual and setpoint pressure of the carrier gas. The range is On/Off; 5–1000 kPa (0.725-145 psi; 0.05-10 bar). This line is not editable when the Constant Flow or Programmed Flow mode is selected.

**Column Flow** — Defines the carrier gas flow rate through the column. The range is On/Off; 0.01-100 mL/min. Select **On** to display the actual and setpoint values. Select **Off** or **0** to turn off all inlet flows. This line is not editable when the Constant Pressure or Programmed Pressure mode is selected.

**Flow Mode** — Defines the carrier gas control mode to use. Each mode activates or deactivates the dedicated parameters.

• **Constant Flow** — The column flow is kept constant throughout the analysis. The pressure at the column head will change with the column temperature to maintain a consistent flow.

- **Constant Pressure** The pressure at the column head is kept constant throughout the analysis. During a temperature program, the column flow decreases due to the increase of the carrier gas viscosity.
- **Programmed Flow** The column flow rate can be programmed to change during the analytical run for up to three flow ramps.

The parameters are:

- **Initial Flow** Defines the beginning flow rate.
- Initial Time Defines how long the Initial Flow is maintained.
- Ramp 1 The ramp rate in mL/min<sup>2</sup> to reach the final flow rate. Select On to enable the ramp and display the setpoint value.
- Final Flow The final flow rate the carrier gas will reach at the end of the ramp rate.
- Final Time Defines how long the corresponding Final flow must be maintained.
- Ramp 2-3 To program additional ramps, select On and enter the ramp rates in mL/min<sup>2</sup>. The Final Flow and Final Time menu items for the ramp are displayed. The ranges and functions of these menu items are identical to the Final Flow and Final Time menu items for Ramp 1.
- **Programmed Pressure** The inlet pressure can be programmed to change during the analytical run up to three pressure ramps.

The parameters are:

- Initial Pressure Defines the initial pressure.
- Initial Time Defines how long the Initial Pressure is maintained.
- Ramp 1 Defines the ramp pressure in kPa/min to reach the Final Pressure. Select
   On to enable the ramp and display the setpoint value.
- Final Pressure Defines the final pressure the carrier gas will reach at the end of the ramp rate.
- Final Time Defines how long the corresponding final pressure must be maintained.
- Ramp 2-3 To program additional ramps, select On and enter the ramp rates in kPa/min. The Final Pressure and Final Time menu items for the ramp are displayed. The ranges and functions of these menu items are identical to the Final Pressure and Final Time menu items for Ramp 1.

**Linear Velocity** — The calculated velocity of the carrier gas through the column, expressed in cm/s. It is not editable.

**Void Time** — The elution time of an un-retained peak, expressed in seconds. It is not editable.

**Gas Saver** — This function reduces carrier gas consumption. The range is On/Off; 5–500 mL/min. Select **On** to turn on the gas saver flow and display the setpoint values. Select **Off** to turn off the gas saver flow. The flow is retained in memory.

**Gas Saver Time** — Defines the time in the run when the gas saver function starts to operate. Usually it starts after the injection to conserve gas. Set a value from 0.00 to 999.99 min. This line does not appear if Gas saver flow is Off.

**Vacuum Comp.** — Use this parameter only when the TRACE 1300/TRACE 1310 is coupled with a mass detector to compensate for vacuum column outlet. The range is On/Off.

# **Injection Mode**

Choose which injection mode to use with the SSLBKF injector. Each mode activates or deactivates the dedicated parameters.

**Split** — The carrier flow is split in the injection port with the bulk going out the split vent. The split vent remains open all the time.

**Splitless** — Closes the split vent during injection to drive the entire sample into the column. Splitless times of about 1 minute are typical.

**Surged Splitless** — Same as **Splitless** but can also program a surge during an injection. Surge starts at Prep Run and continues until the surge duration time is finished. Surge is further defined in next group of controls.

# **Inlet Parameters**

The SSLBKF injection parameters are described below:

**Temperature** — Defines the setpoint for the injector's temperature. Depending on the injection mode you select, set the temperature high enough to vaporize the sample and solvent. Enter a value for the inlet temperature in the range of 0-400 °C.

**Split Flow** — Enter a value in the range of 5-1250 mL/min. The **Split Ratio** is adjusted automatically. In addition, this value is governed by the initial column flow rate entered on the associated carrier gas control. If the flow rate is changed, the Split Flow value is adjusted so that the Split Ratio is maintained. However, if the Split Flow value then falls outside its limits, a warning is generated.

**Split Ratio** — Enabled when the injection mode is set to **Split**. It is also associated to the Flow Mode set to either Constant Flow or Programmed Flow. Specify the ratio of split flow to column flow. Calculate the split ratio: Split Ratio = (split flow) / (column flow). Enter a value in the range of 1-12500. The Split Flow entry is adjusted automatically.

**Splitless Time** — Enabled when the injection mode is set to either **Splitless** or **Surged Splitless**. Specify the length of time the split valve remains closed after a splitless injection. Enter a value in the range of 0.00-999.99 min. The timer begins at the start of the run. The split vent reopens when the splitless time ends.

# **Purge Parameters**

Purge parameters are available when the injection Mode is set to either **Splitless** or **Surged Splitless**.

**Constant Septum Purge** — Controls the septum purge for the injector. The range is On; Off. Select **On** to activate the function to continuously flush the septum with a purge flow.

**Note** The purge flow, in the range 0.5-50 mL/min, must be set in the Configuration Page of the injector module.

**Stop Purge For** — Enabled if the Constant Septum Purge parameter is set to **On**. You can then enter a time from 0.00 to 999.99 min at which the septum purge ceases.

## **Surge Parameters**

Surge parameters are enabled when the injection Mode is set to Surged Splitless.

**Surge Pressure** — Defines the pressure applied during the splitless time to produce a surge of flow in the injector to speed transfer of the sample. Enter a value in the range of 5-1000 kPa (0.725-145 psi; 0.05-10 bar).

**Surge Duration** — Defines the time that the surge pressure is maintained. Enter a value in the range of 0.00-999.99 min. Typically, set to coincide with the **Splitless time**.

# **Setting Gas Parameters**

Before starting, check that the carrier gas type is correct for the analysis.

#### To set gas parameters

- 1. Program the carrier gas flow.
  - a. Select Flow Mode.
  - b. Choose the mode you want choosing among Constant flow, Constant Pressure, Programmed Flow or Programmed Pressure.
  - c. Enter the initial Flow or Pressure.
    - i. If you select **Constant Flow** mode, enter the desired **Column Flow** value. The pressure necessary is calculated and adjusted to maintain the constant flow.
    - ii. If you select Constant Pressure mode, enter the desired Pressure value.

d. Enter a **Programmed Flow/Programmed Pressure**.

**Note** When you select Programmed Flow or Programmed Pressure mode, the menu for the carrier contains parameters for up to three program ramps.

- i. Select **Progr Flow/Pressure**, scroll to Initial Flow/Press and enter the desired value. Press Enter.
- ii. Scroll to Initial Time and enter a value. This parameter ends the initial part of the program.
- e. Program the Ramps.
  - i. Scroll to Ramp 1 and enter the value.
  - ii. Scroll to Final Flow 1/Pressure 1 and enter the final value for the ramp.
  - iii. Scroll to Final Time 1 and enter the final time for Ramp 1. This operation ends the first ramp setting.
  - iv. If you do not want a second ramp, leave Ramp 2 set to **Off**. To enter a second ramp, scroll to Ramp 2 and enter the value.
  - v. Scroll to Final Flow 2/Pressure 2 and enter the final value for the ramp.
  - vi. Scroll to Final Time 2 and enter the final time for Ramp 2. This operation ends the second ramp setting.
  - vii. If you do not want a third ramp, leave Ramp 3 set to **Off**. To enter a third ramp, scroll to Ramp 3 and enter the value.
  - viii. Scroll to Final Flow 3/Pressure 3 and enter the final value for the ramp.
  - ix. Scroll to Final Time 3 and enter the final time for Ramp 3. This operation ends the third ramp setting.
- 2. If the TRACE 1300/TRACE 1310 is working with a mass detector, set **Vacuum Compensation** to **On** to compensate for vacuum column outlet.

## **Setting Parameters for Backflush**

Use the following procedure to setup a Backflush injection. In this mode the heavier part of the sample is diverted out to the split line, preserving the analytical column.

Before stating, verify that the correct liner is installed into the injector body and the system is free of leaks.

#### To set parameters for backflush

- 1. Program the carrier gas flow.
- 2. From the mode list, choose Splitless
- 3. Set the injector **Temperature**.

- 4. Enter the desired **Split Flow** value.
- 5. Enter the **Splitless Time**.
- 6. If desired turn on **Constant Septum Purge** and set in **Stop Purge For** how many minutes elapse off before restarting the purge.
- 7. Enable backflush.
- 8. Set the time at which the backflush valve is activated.
- 9. Set the backflush duration (Specific time or GC runtime).
- 10. If Specific time has been selected, specify the backflush duration.

# Setting Parameters for the Split Mode

Before starting, verify that the correct liner is installed into the injector body and the system is free of leaks.

#### \* To set a Split injection

- 1. Program the carrier gas flow.
- 2. From the mode list, choose **Split**.
- 3. Set the injector **Temperature**.
- 4. If carrier **Flow** Mode (Programmed or Constant) is selected, specify the Split Flow or Split Ratio.
  - a. If you want a specific Split Flow, enter that value. Split Ratio will be calculated.
  - b. If you want a specific Split Ratio, enter that value. Split Flow will be calculated.
- 5. If desired, turn on Gas Saver and set Gas Saver Time after the injection time.

# **Setting Parameters for the Splitless Mode**

Before starting, verify that the correct liner is installed into the injector body and the system is free of leaks.

#### To set a Splitless injection

- 1. Program the carrier gas flow.
- 2. From the mode list, choose **Splitless**.
- 3. Set the injector Temperature.
- 4. Enter the **Splitless Time**.
- 5. If desired turn on **Constant Septum Purge** and set in **Stop Purge For** how many minutes elapse before restarting the purge.

6. If desired, turn on Gas Saver and set Gas Saver Time after the injection time.

# **Setting Parameters for the Surged Splitless Mode**

Before starting verify that the correct liner is installed into the injector body and the system is free of leaks.

#### ✤ To set a Surged Splitless injection

- 1. Program the carrier gas flow.
- 2. From the Mode list, choose **Surged Splitless**.
- 3. Set the injector Temperature.
- 4. Enter the **Splitless Time**.
- 5. If desired turn on **Constant Septum Purge** and set in **Stop Purge For** how many minutes elapse before restarting the purge.
- 6. Set the values for Surge Pressure and Surge Duration.
- 7. If desired, turn on Gas Saver and set Gas Saver Time after the injection time.

# **Connecting the SSL Backflush System**

For connecting the SSL Backflush System into the oven of the GC refer to the section **Connecting the SSL/PTV Backflush System** in the *TRACE 1300/TRACE 1310 Hardware Manual*.

# 8

# **Gas Sampling Valve (GSV) Module**

This chapter describes the Gas Sampling Valve (GSV) module and provides instructions for programming the inlet parameters.

#### Contents

- Module Overview
- GSV Injection
- Backflush Mode
- Consumables
- Using GSV Parameters
- Connecting the Backflush System

# **Module Overview**

The Gas Sampling Valve module includes a 6-port diaphragm valve, heater body, digital pneumatics for the control of the gases, backflush line, and fittings for the connection to the analytical column.

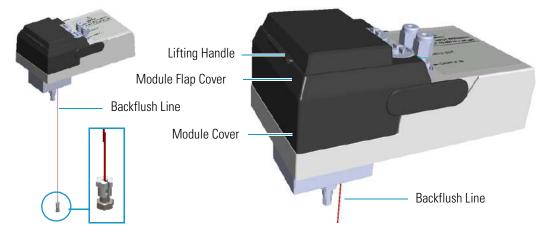


Figure 98. Gas Sampling Valve Module

The components of the module are shown in Figure 99.

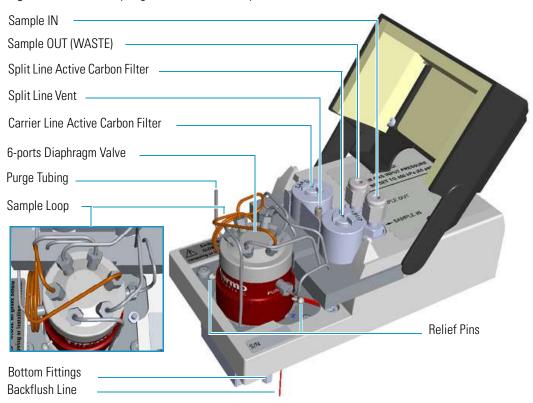
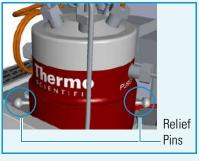


Figure 99. Gas Sampling Valve Module Components

**IMPORTANT** The valve is delivered with relief pins. These pins MUST BE removed from the valve body before working with the valve.





Once actuation tubing has been set up, and pressure has been adjusted to 450 kPa (65 psig), actuate the valve and remove the pins. You may need to increase the pressure a little to ease hand removal of the pins. Remember, make sure to properly readjust the actuating operating pressure after removing the pins, if you had to change it. Keeps these pins in a safe place. You may want to re-use them for valve maintenance. It is good practice to re-install the relief

pins if the valve is not used for a long time. For details refer to the instructions provided by the valve manufacturer.

**WARNING** DO NOT SET THE CARRIER GAS INPUT PRESSURE HIGHER THAN 450 kPa (65 psig; 4.5 bar), OTHERWISE THE VALVE MAY BE DAMAGED.



**WARNING** DO NOT USE HYDROGEN AS CARRIER GAS. The module is not compatible with Hydrogen carrier gas. The same gas used as carrier gas is used to purge and to actuate the valve and must be an inert gas.

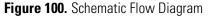
# **GSV** Injection

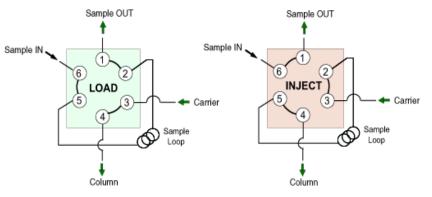
In quantitative gas chromatographic determination of a gas mixture, the sample must be introduced in reproducible amounts. This device allows the introduction of a precise amount of gas sample directly into the chromatographic capillary column with a simple rapid operation.

A wide range of sampling loops allows the injections of different volume of samples. The sampling loop is installed between the ports 2 and 5 of the gas sampling valve.

The switching from load sample to inject sample position (and vice-versa) is controlled through the user interface. Load is the default switching position. In Load position the valve is On, in Inject position the valve is OFF.

Figure 100 shows the schematic flow diagram of the gas sampling valve.





# **Backflush Mode**

This operating mode allows elimination of the analytes in the sample not relevant for the analysis.

# **Consumables**

The consumables required for this module are the sampling loop. The following volume calibrated sampling loop are available:  $20 \ \mu$ L,  $50 \ \mu$ L,  $100 \ \mu$ L,  $250 \ \mu$ l as standard,  $500 \ \mu$ L, and  $1000 \ \mu$ L.

# **Using GSV Parameters**

This menu includes the operating parameters for the Gas Sampling Valve module. Editable parameters vary with the **operating mode: split, splitless, surged splitless**, and the **flow mode: constant flow, constant pressure, programmed flow, programmed pressure**. You can also perform backflush applications.

- Carrier Gas Parameters
- Injection Mode
- Inlet Parameters
- Surge Parameters
- Valve Parameters
- Setting Gas Parameters
- Setting Parameters for Backflush
- Setting Parameters for the Split Mode
- Setting Parameters for the Splitless Mode
- Setting Parameters for the Surged Splitless Mode

The following sections list and describe the parameters controlling the front/back GSV injector. They are:

## **Carrier Gas Parameters**

Set the carrier gas control parameters. Visualized parameters change according to the **Flow Mode** set.

**Pressure** — Defines the actual and setpoint pressure of the carrier gas. The range is On/Off; 5–1000 kPa (0.725-145 psi; 0.05-10 bar). This line is not editable when the Constant Flow or Programmed Flow mode is selected.

**Column Flow** — Defines the carrier gas flow rate through the column. The range is On/Off; 0.01-100 mL/min. Select **On** to display the actual and setpoint values. Select **Off** or **0** to turn off all inlet flows. This line is not editable when the Constant Pressure or Programmed Pressure mode is selected.

**Flow Mode** — Defines the carrier gas control mode to use. Each mode activates or deactivates the dedicated parameters.

• **Constant Flow** — The column flow is kept constant throughout the analysis. The pressure at the column head will change with the column temperature to maintain a consistent flow.

- **Constant Pressure** The pressure at the column head is kept constant throughout the analysis. During a temperature program, the column flow decreases due to the increase of the carrier gas viscosity.
- **Programmed Flow** The column flow rate can be programmed to change during the analytical run for up to three flow ramps.

The parameters are:

- **Initial Flow** Defines the beginning flow rate.
- Initial Time Defines how long the Initial Flow is maintained.
- Ramp 1 The ramp rate in mL/min<sup>2</sup> to reach the final flow rate. Select On to enable the ramp and display the setpoint value.
- Final Flow The final flow rate the carrier gas will reach at the end of the ramp rate.
- Final Time Defines how long the corresponding Final flow must be maintained.
- Ramp 2-3 To program additional ramps, select On and enter the ramp rates in mL/min<sup>2</sup>. The Final Flow and Final Time menu items for the ramp are displayed. The ranges and functions of these menu items are identical to the Final Flow and Final Time menu items for Ramp 1.
- **Programmed Pressure** The inlet pressure can be programmed to change during the analytical run up to three pressure ramps.

The parameters are:

- **Initial Pressure** Defines the initial pressure.
- Initial Time Defines how long the Initial Pressure is maintained.
- Ramp 1 Defines the ramp pressure in kPa/min to reach the Final Pressure. Select
   On to enable the ramp and display the setpoint value.
- Final Pressure Defines the final pressure the carrier gas will reach at the end of the ramp rate.
- Final Time Defines how long the corresponding final pressure must be maintained.
- Ramp 2-3 To program additional ramps, select On and enter the ramp rates in kPa/min. The Final Pressure and Final Time menu items for the ramp are displayed. The ranges and functions of these menu items are identical to the Final Pressure and Final Time menu items for Ramp 1.

**Linear Velocity** — The calculated velocity of the carrier gas through the column, expressed in cm/s. It is not editable.

**Void Time** — The elution time of an un-retained peak, expressed in seconds. It is not editable.

**Gas Saver** — This function reduces carrier gas consumption. The range is On/Off; 5–500 mL/min. Select **On** to turn on the gas saver flow and display the setpoint values. Select **Off** to turn off the gas saver flow. The flow is retained in memory.

**Gas Saver Time** — Defines the time in the run when the gas saver function starts to operate. Usually it starts after the injection to conserve gas. Set a value from 0.00 to 999.99 min. This line does not appear if Gas saver flow is Off.

**Vacuum Comp.** — Use this parameter only when the TRACE 1300/TRACE 1310 is coupled with a mass detector to compensate for vacuum column outlet. The range is On/Off.

# **Injection Mode**

Choose which injection mode to use with the GSV inlet. Each mode activates or deactivates the dedicated parameters.

**Split** — The carrier flow is split in the injection port with the bulk going out the split vent. The split vent remains open all the time. This mode enables the **Valve** parameters.

**Splitless** — Closes the split vent during injection to drive the entire sample into the column. The **Valve** parameters are disabled. In this case the sampling time (injection duration) of the gas sampling valve coincides with the splitless time. Splitless times of about 1 minute are typical.

**Surged Splitless** — Same as **Splitless** but can also program a surge during an injection. Surge starts at Prep Run and continues until the surge duration time is finished. Surge is further defined in next group of controls. The **Valve** parameters are disabled. In this case the sampling time (injection duration) of the gas sampling valve coincides with the splitless time.

# **Inlet Parameters**

The GSV injection parameters are described below:

**Temperature** — Defines the setpoint for the gas sampling valve temperature. Enter a value in the range of 0-150 °C.

**Split Flow** — Enter a value in the range of 5-1250 mL/min. The **Split Ratio** is adjusted automatically. In addition, this value is governed by the initial column flow rate entered on the associated carrier gas control. If the flow rate is changed, the Split Flow value is adjusted so that the Split Ratio is maintained. However, if the Split Flow value then falls outside its limits, a warning is generated.

**Split Ratio** — Enabled when the injection mode is set to **Split**. It is also associated to the Flow Mode set to either Constant Flow or Programmed Flow. Specify the ratio of split flow to column flow. Calculate the split ratio: Split Ratio = (split flow) / (column flow). Enter a value in the range of 1-12500. The Split Flow entry is adjusted automatically.

**Splitless Time** — Enabled when the injection mode is set to either **Splitless** or **Surged Splitless**. Specify the length of time the split valve remains closed after a splitless injection. The splitless time coincides with the **injection duration** parameter of the gas sampling valve. Enter a value in the range of 0.00-999.99 min. The timer begins at the start of the run. The split vent reopens when the splitless time ends.

# **Surge Parameters**

Surge parameters are enabled when the injection Mode is set to Surged Splitless.

**Surge Pressure** — Defines the pressure applied during the splitless time to produce a surge of flow in the injector to speed transfer of the sample. Enter a value in the range of 5-1000 kPa (0.725-145 psi; 0.05-10 bar).

**Surge Duration** — Defines the time that the surge pressure is maintained. Enter a value in the range of 0.00-999.99 min. Typically, set to coincide with the **Splitless time**.

## **Valve Parameters**

Valve parameters are enabled when the injection Mode is set to Split.

Injection start time — Defines the time at which the injection must begin.

**Injection duration** — Defines the time the sampling valve must be maintained on injection position.

# **Setting Gas Parameters**

Before starting, check that the carrier gas type is correct for the analysis.

#### To set gas parameters

- 1. Program the carrier gas flow.
  - a. Select Flow Mode.
  - b. Choose the mode you want choosing among Constant flow, Constant Pressure, Programmed Flow or Programmed Pressure.
  - c. Enter the initial **Flow** or **Pressure**.
    - i. If you select **Constant Flow** mode, enter the desired **Column Flow** value. The pressure necessary is calculated and adjusted to maintain the constant flow.
    - ii. If you select **Constant Pressure** mode, enter the desired **Pressure** value.
  - d. Enter a **Programmed Flow/Programmed Pressure**.

**Note** When you select Programmed Flow or Programmed Pressure mode, the menu for the carrier contains parameters for up to three program ramps.

- i. Select **Progr Flow/Pressure**, scroll to Initial Flow/Press and enter the desired value. Press Enter.
- ii. Scroll to Initial Time and enter a value. This parameter ends the initial part of the program.
- e. Program the **Ramps**.
  - i. Scroll to Ramp 1 and enter the value.
  - ii. Scroll to Final Flow 1/Pressure 1 and enter the final value for the ramp.
  - iii. Scroll to Final Time 1 and enter the final time for Ramp 1. This operation ends the first ramp setting.
  - iv. If you do not want a second ramp, leave Ramp 2 set to **Off**. To enter a second ramp, scroll to Ramp 2 and enter the value.
  - v. Scroll to Final Flow 2 /Pressure 2 and enter the final value for the ramp.
  - vi. Scroll to Final Time 2 and enter the final time for Ramp 2. This operation ends the second ramp setting.
  - vii. If you do not want a third ramp, leave Ramp 3 set to **Off**. To enter a third ramp, scroll to Ramp 3 and enter the value.
  - viii. Scroll to Final Flow 3/Pressure 3 and enter the final value for the ramp.
  - ix. Scroll to Final Time 3 and enter the final time for Ramp 3. This operation ends the third ramp setting.
- 2. If the TRACE 1300/TRACE 1310 is working with a mass detector, set **Vacuum Compensation** to **On** to compensate for vacuum column outlet.

## **Setting Parameters for Backflush**

Use the following procedure to setup a Backflush injection. In this mode the heavier part of the sample is diverted out to the split line, preserving the analytical column.

Before stating, verify that the correct liner is installed into the injector body and the system is free of leaks.

#### To set parameters for backflush

- 1. Program the carrier gas flow.
- 2. From the mode list, choose Splitless or Split.
- 3. Set the gas sampling valve **Temperature**.
- 4. Enter the desired **Split Flow** value.
- 5. Enter the **Splitless Time** (in case of Splitless mode; see the point 2).
- 6. Enable backflush.

- 7. Set the time at which the backflush valve is activated.
- 8. Set the backflush duration (Specific time or GC runtime).
- 9. If Specific time has been selected, specify the backflush duration.

## **Setting Parameters for the Split Mode**

Before starting, verify that the correct liner is installed into the injector body and the system is free of leaks.

#### To set a Split injection

- 1. Program the carrier gas flow.
- 2. From the mode list, choose Split.
- 3. Set the gas sampling valve Temperature.
- 4. If carrier **Flow** Mode (Programmed or Constant) is selected, specify the Split Flow or Split Ratio.
  - a. If you want a specific Split Flow, enter that value. Split Ratio will be calculated.
  - b. If you want a specific Split Ratio, enter that value. Split Flow will be calculated.
- 5. Set the **Injection start time** and the **Injection duration** for the gas sampling valve.
- 6. If desired, turn on Gas Saver and set Gas Saver Time after the injection time.

## **Setting Parameters for the Splitless Mode**

Before starting, verify that the correct liner is installed into the injector body and the system is free of leaks.

- To set a Splitless injection
- 1. Program the carrier gas flow.
- 2. From the mode list, choose **Splitless**.
- 3. Set the gas sampling valve Temperature.
- 4. Enter the **Splitless Time**.
- 5. If desired, turn on Gas Saver and set Gas Saver Time after the injection time.

# Setting Parameters for the Surged Splitless Mode

Before starting verify that the correct liner is installed into the injector body and the system is free of leaks.

#### ✤ To set a Surged Splitless injection

- 1. Program the carrier gas flow.
- 2. From the Mode list, choose **Surged Splitless**.
- 3. Set the gas sampling valve Temperature.
- 4. Enter the **Splitless Time**.
- 5. Set the values for Surge Pressure and Surge Duration.
- 6. If desired, turn on Gas Saver and set Gas Saver Time after the injection time.

# **Connecting the Backflush System**

For connecting the Backflush System of the Gas Sampling Valve module into the oven of the GC, refer to the section **Connecting a GSV Backflush System** in the *TRACE 1300/TRACE 1310 Hardware Manual*.

Instant Connect Helium Saver Injector Module (HeS-S/SL)

This chapter describes the Instant Connect Helium Saver Injector Module (HeS-S/SL) and provides instructions for programming the inlet parameters.

#### Contents

- Module Overview
- Principle of Operation
- Gas Requirements
- Operation
- Optimizing Helium Conservation
- Consumables
- Using Instant Connect Helium Saver Injector Module Parameters

# **Module Overview**

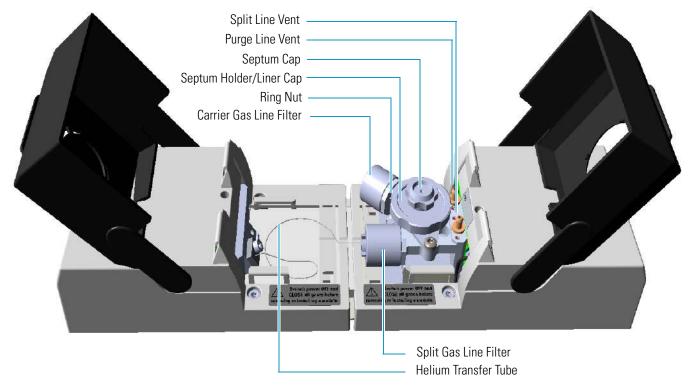
The Instant Connect Helium Saver Injector Module "double wide" module is an innovative split/splitless (S/SL) inlet system capable of dramatically reducing consumption of increasingly expensive and difficult-to-procure helium gas. In addition, the detrimental effects of continual out gassing and deposition of matrix residuals onto the head of the column are reduced. This may prolong column lifetime, reduce the frequency of column trimmings, and improve analytical performance.

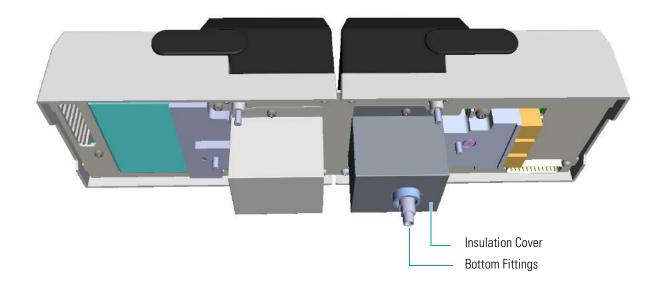
Figure 101. Instant Connect Helium Saver Injector Module



The module and injector components are shown in Figure 102 and Figure 103.

#### Figure 102. Instant Connect Helium Saver Injector Module Components





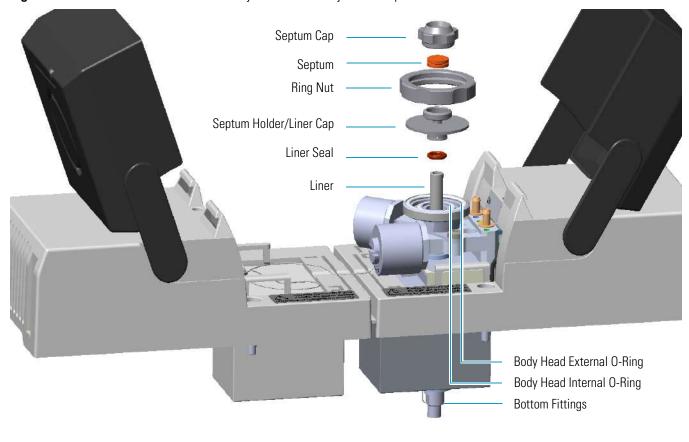


Figure 103. Instant Connect Helium Saver Injector Module: Injector Components



**READ MANUAL** Refer also to the *TRACE 1300/TRACE 1310 Hardware Manual* and to *Instant Connect Saver Injector Module User Guide (PN 31709737)*.

# **Principle of Operation**

In a conventional split/splitless injector, the amount of carrier gas used for analytical separation is typically on the order of 1 milliliter per minute (mL/min), while the split flow plus the septum purge flow may be as high as 50 times this amount. The novel module essentially eliminates large helium consumption by decoupling the gas used for the analysis from the gas used for maintaining split and purge flows. The split and purge flows are accomplished using nitrogen, while the carrier gas remains helium. This allows for all the advantages of helium operation with mass spectrometers (inertness, sensitivity, and safety) while at the same time conserving helium, a limited natural resource. Using a laboratory nitrogen gas generator with the module also allows for reducing the frequency of high-pressure cylinder changes from months to years.

Figure 104 on page 178 illustrates the injector module in the default mode of operation.

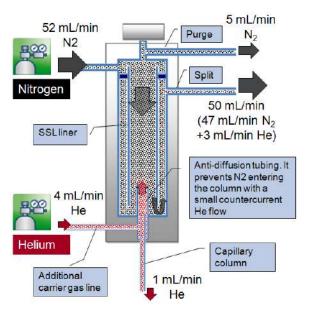
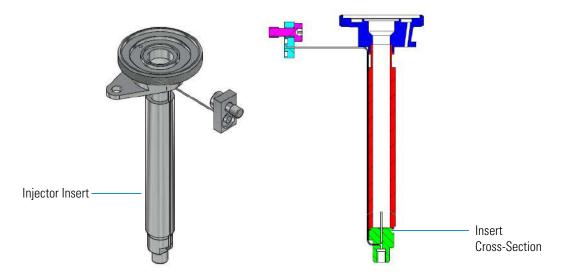


Figure 104. Instant Connect Helium Saver Injector Module in Default Mode

Nitrogen gas enters near the top of the inlet in the same manner as a conventional SSL and pressurizes the inlet. The nitrogen flow is caused to flow down the bore of a glass liner, then around the outer diameter and out a split vent located near the top of the injector.

A small-bore stainless-steel tube runs along the side of the injector body in order to preheat a small helium flow. The small helium flow is selectable between two levels, typically 4 mL/min or 0.1 mL/min based on the setting of a valve contained in a compartment to the left of the inlet housing that uses fixed capillary restrictors and a set head pressure. The helium flow is delivered into the base of the heated injector body at a junction near the tip of the analytical column, but before a short segment of tubing that acts as a back diffusion barrier. The insert details are illustrated in Figure 105.

Figure 105. Instant Connect Helium Saver Injector Module Insert



The inlet is pressurized with nitrogen, but when the helium delivery is set to 4 mL/min (the default condition), the column consumes 1 mL/min of helium (if that is the desired flow rate), while the 3 mL/min excess is diverted upward, contributing to the bulk nitrogen gas purge in the confines of the injector body. The 3 mL/min of upward helium flow is sufficient to prevent significant back diffusion of nitrogen into the carrier gas stream. In the default condition, matrix residuals residing in the injection port liner are precluded from entering the analytical column. During an injection sequence, (illustrated in Figure 106) the helium flow is switched to 0.1 mL/min, which acts as a residual purge to keep the gas delivery line free from injected solvents and contamination.

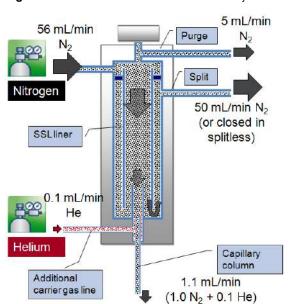


Figure 106. Instant Connect Helium Saver Injector Module Injection Mode

During this time, nitrogen acts to sweep the injected analytes onto the column for the duration of the splitless time when in splitless mode, or for a few seconds (the helium delay time) when in split mode. The short (12 mm) back diffusion barrier offers very low flow restriction and residence time for analytes, resulting in identical retention times and peak shapes. The low residence time of analytes in the back diffusion barrier as well as a proprietary inert coating also ensures no activity toward fragile or surface active analytes.

# **Gas Requirements**

Nitrogen 99.999% with a gas regulator output capable of up to 150 psig (1030 kPa; 10.3 bar).

The nitrogen supply should be attached to a high capacity oxygen trap designed for carrier gas purification. Reactive ambient temperature traps such as Restek<sup>TM</sup> #20601 are suitable. This trap has the capacity to purify more than five 200 ft<sup>3</sup> (37 m<sup>3</sup>) cylinders to 15 pbb when the incoming gas purity is 99.999%.

Helium 99.999% with a gas regulator output capable of up to 150 psig (1030 kPa; 10.3 bar).

Helium purity is critically important, particularly with respect to oxygen, nitrogen, and water vapor. Conventional chemical traps at low helium flow rates add contamination rather than remove it, particularly regarding nitrogen and argon.

It is highly recommended that you use a heated zirconium-based gettering trap specifically designed for helium purification. These gettering traps efficiently remove nitrogen, which is difficult to remove with conventional chemical traps. Thermo Scientific part number 1R120577-0001 contains all the materials necessary for treatment of nitrogen and helium supplies.

The helium should be delivered using the stainless steel tubing provided with the purification kit, with the tubing connections leading directly from the gettering trap to the GC make-up gas connection. The gettering trap should be located immediately adjacent to the GC. The standard outfit kit contains a 1 m length of 1/16-in. stainless steel tubing which can be used along with a customer supplied gettering trap if desired. Remote cylinder placement is acceptable provided the heated gettering trap is located adjacent to the GC using small bore tubing. Internal surface area should be minimized in order to reduce water vapor intensity in the air/water spectrum of a GC/MS.

# Operation

#### To operate the Instant Connect Helium Saver Injector Module

- 1. Turn on the GC power and establish nitrogen flow to the inlet by setting a carrier gas flow, split flow, and septum purge flow as for a standard SSL injector.
- 2. Ensure the inlet has been configured with the conservation enabled. This will establish approximately 4 mL/min of helium flow to the back diffusion preventer of the inlet when the helium pressure is 110 psig (760 kPa; 7.6 bar) at the regulator.

**Note** Table 11 serves as a guide for setting the correct helium regulator pressure.

**Table 11.** Determining Correct Helium Regulator Pressure for Column Type (Sheet 1 of 2)

Column Length	Column i.d.	Desired Flow <sup>1</sup>	Required helium regulator pressure psig (kPa; bar) <sup>2</sup>
5 m/10 m	0.10 mm	0.4 mL/min	110 psig (760 kPa; 7.6 bar)
		0.5 mL/min	120 psig (830 kPa; 8.3 bar)
		0.6 mL/min	130 psig (900 kPa; 9 bar)
10 m/20 m	0.18 mm	0.8 mL/min	100 psig (690 kPa; 6.9 bar)
		1.0 mL/min	110 psig (760 kPa; 7.6 bar)
		1.2 mL/min	120 psig (830 kPa; 8.3 bar)
		1.5 mL/min	130 psig (900 kPa; 9 bar)
		2.0 mL/min	140 psig (970 kPa; 9.7 bar)
15 m	0.25 mm	1.0 mL/min	100 psig (690 kPa; 6.9 bar)

		•	
Column Length	Column i.d.	Desired Flow <sup>1</sup>	Required helium regulator pressure psig (kPa; bar) <sup>2</sup>
		1.2 mL/min	110 psig (760 kPa; 7.6 bar)
		1.7 mL/min	120 psig (830 kPa; 8.3 bar)
		2.2 mL/min	130 psig (900 kPa; 9 bar)
		2.7 mL/min	140 psig (970 kPa; 9.7 bar)
30 m	0.25 mm	1.0 mL/min	100 psig (690 kPa; 6.9 bar)
		1.2 mL/min	110 psig (760 kPa; 7.6 bar)
		1.7 mL/min	120 psig (830 kPa; 8.3 bar)
		2.2 mL/min	130 psig (900 kPa; 9 bar)
		2.7 mL/min	140 psig (970 kPa; 9.7 bar)
		3.2 mL/min	150 psig (1030 kPa; 10.3 bar)
60 m	0.25 mm	1.0 mL/min	120 psig (830 kPa; 8.3 bar)
		1.2 mL/min	130 psig (900 kPa; 9 bar)
		1.7 mL/min	140 psig (970 kPa; 9.7 bar)
		2.2 mL/min	150 psig (1030 kPa; 10.3 bar)
100 m	0.25 mm	1.0 mL/min	130 psig (900 kPa; 9 bar)
		1.2 mL/min	140 psig (970 kPa; 9.7 bar)
		1.7 mL/min	150 psig (1030 kPa; 10.3 bar)
30 m	0.32 mm	1.5 mL/min	100 psig (690 kPa; 6.9 bar)
		2.0 mL/min	110 psig (760 kPa; 7.6 bar)
		2.5 mL/min	120 psig (830 kPa; 8.3 bar)
		3.0 mL/min	130 psig (900 kPa; 9 bar)
		3.5 mL/min	140 psig (970 kPa; 9.7 bar)
60 m	0.32 mm	1.5 mL/min	110 psig (760 kPa; 7.6 bar)
		2.0 mL/min	120 psig (830 kPa; 8.3 bar)
		2.5 mL/min	130 psig (900 kPa; 9 bar)
		3.0 mL/min	140 psig (970 kPa; 9.7 bar)
		3.5 mL/min	150 psig (1030 kPa; 10.3 bar)
100 m	0.32 mm	1.5 mL/min	130 psig (900 kPa; 9 bar)
		2.0 mL/min	140 psig (970 kPa; 9.7 bar)
		2.5 mL/min	150 psig (1030 kPa; 10.3 bar)

Table 11. Determining Correct Helium Regulator Pressure for Column Type (Sheet 2 of 2)

<sup>1</sup> For flow rates not specifically listed, round up in pressure to the next highest value. For example, if 1.5 mL/min is desired using a 0.25 mm ID column of 15 m length, use a helium regulator pressure of 110 psig (760 kPa; 7.6 bar).

 $^2$  Minimum pressure required to avoid nitrogen back diffusion into the helium carrier gas. Higher pressure will result in a slightly higher (than minimum) consumption of helium, but will not result in adverse analytical performance.

- 3. Interface the terminal end of the GC column to the mass spectrometer and pump the system down. Do not enable the thermal zones at this point.
- 4. After several minutes, check the air/water spectrum if the system appears relatively leak free (the system has good ion gauge pressure). There likely will be some residual nitrogen and water vapor in the gas lines which may take overnight to subside.
- 5. If the air/water spectrum otherwise looks good, heat the ion source, transfer line and injector to their respective operational temperatures.
- 6. Tune the mass spectrometer according to standard procedure. The autotune should appear normal and report a passing leak check.
- 7. The Instant Connect Helium Saver Injector Module operates identically to the standard SSL with respect to setting column flows, pressures, temperatures, and other features. It is best to leave the "Enable Conservation" field of the inlet configuration defaulted to Yes. This will ensure that nitrogen is delivered to the GC column only during the splitless time for splitless injections.
- 8. It is also necessary to configure the Oven "**Ready Delay**" for 1 minute. This will allow the GC column to completely fill with nitrogen before an injection is made, as well as serve to allow the residual purge to stabilize at a low value.

## **Split Mode Injections**

If the injection is a Split mode injection, some of the sample will enter the column and the remainder will exit the split vent in accordance with the split ratio. The split is accomplished using nitrogen, but it is necessary to switch to helium following the injection. If the switch to helium is done at the same time as the injection, the sample will be largely lost, being occluded from entering the column by the back flow of helium. A few seconds are needed for the sample transfer to the column, particularly for heavy molecular weight components. This delay is called the **Helium delay** in the inlet configuration menu, and a value of 0.10 minutes should be sufficient time for sample transfer even for heavy components.

#### **Maximizing Helium Lifetime**

It is possible to extend the lifetime of a cylinder of helium beyond three years of continuous use to more than ten by putting the inlet in a standby condition when the GC is not in use. This is done by configuring the **Enable conservation** field to read **No**. This puts the helium delivery valve in the residual purge mode, which allows nitrogen to enter the column continuously. It is suggested that this mode of operation only be attempted after complete familiarization of the inlet and its operation.

Also note that water vapor will accumulate in the gas lines when the GC is in standby condition. For this reason, operation should not commence until a few hours after reconfiguring the **Enable conservation** field to **Yes**.

Do not attempt to tune the MS when **Enable conservation** is set to **No**. For most situations, it is best to leave **Enable conservation** set to **Yes**. This eliminates any waiting for water vapor to subside and still results in good cylinder lifetime.

# **Optimizing Helium Conservation**

The helium flow delivered to the back diffusion preventer (tubing) on the base of the SSL is of two different flow rates as mentioned in "Principle of Operation" on page 177. The default flow is approximately 4 mL/min for all times except during injection, when it is reduced to approximately 0.1 mL/min. Since the flow is delivered by setting a pressure upstream of a capillary restrictor designed into the module, the actual flow rates depend on the inlet pressure as well as the input helium pressure.

For a 30 m column of 0.25 mm i.d. and 1.2 mL/min column flow rate, the helium pressure should be set to approximately 110 psig (760 kPa; 7.6 bar). This will ensure that nitrogen is occluded from entering the column even at the highest column temperature where the inlet pressure will also be at its highest point. It is important to note that the actual helium flow is not critical to the operation of the inlet since the inlet pressure is set by the nitrogen flow.

An excess of helium will simply be diverted upward into the inlet where it contributes to the bulk purge flow. Since the purpose of the inlet is conserving helium, the reduction of helium to a minimum can be accomplished in the following manner.

- 1. Ensure helium conservation is enabled in the inlet configuration page and that a minute or two has elapsed for any remaining nitrogen to exit the column. Start with 100 psig (690 kPa; 6.9 bar) helium pressure. Nitrogen will give a higher response than helium for both ion gauge equipped systems and systems having only a convection gauge. These gauges can be used to evaluate if nitrogen or helium is exiting the column and can also be used as guides for method development.
- 2. At the initial oven temperature of the GC method, ensure an adequate air/water spectrum. The air/water spectrum should exhibit a nitrogen peak (*m/z* 28) no larger than twice the intensity of water vapor (*m/z* 18). If the nitrogen peak is larger than this, stop scanning the air/water spectrum and increase the regulator pressure to 110 psig (760 kPa; 7.6 bar). Wait a minute for the system to stabilize and again check the air/water spectrum. Repeat these steps until nitrogen does not dominate the spectrum. An autotune should indicate a leak free condition. Do not leave the instrument continually scanning air/water when nitrogen dwarfs the spectrum. This could result in decreased electron multiplier lifetime.
- 3. After getting a good air/water spectrum as in step 2, stop scanning air/water and increase the oven temperature to the final temperature used in the method. Allow a minute for stabilization to occur then check the air/water spectrum again. Ensure the nitrogen peak is of similar intensity to that in step 2.

- 4. If the nitrogen peak dwarfs the intensity of water vapor  $(m/z \ 18)$  and oxygen  $(m/z \ 32)$ , stop scanning and increase the helium pressure at the regulator to 10 psig (69 kPa; 0.69 bar) above the present setting and wait for one minute for the system to stabilize.
- 5. Check the air/water spectrum to be sure the nitrogen is reduced to an acceptable level. Increase the regulator pressure in 10 psig (69 kPa; 0.69 bar) increments if necessary until the nitrogen is reduced.
- 6. Cool the GC oven to the initial oven temperature. Keep the regulator pressure set to that obtained in step 5. This will ensure nitrogen is only a minor contaminant at all points the GC run regardless of the inlet pressure (which increases with oven temperature) for constant flow mode conditions.

## Notes on Air/Water Spectra

The mass spectrometer should pass the leak check diagnostic routine in Auto Tune which evaluates the amount of oxygen present. In general, the intensity of water vapor  $(m/z \ 18)$  should be less than ten times the intensity of oxygen  $(m/z \ 32)$  with a passing leak check in Auto Tune. The amount of water vapor present will depend on how long the system has been in operation, and whether the helium flow has been put into standby condition either overnight or over the course of a weekend. The nitrogen peak  $(m/z \ 28)$  should be greater than two times the indicated abundance of oxygen but should not dwarf the abundance of water vapor.

# Consumables

The consumables required for this injector are the septum, the glass liner, and the o-rings.

#### Septum

You should always use good quality septa, such as the BTO septum supplied with the TRACE 1300/TRACE 1310. Such septa resist deformation, have longer life expectancy, and have a low bleed level, even at high temperatures.

The SSL injector is compatible with the **Merlin Microseal<sup>™</sup> High Pressure Valve** instead of the standard septum.

#### Liner

The sample injection mode used determines the choice of liner to install into the injector body. An appropriate liner must ensure complete sample vaporization and contain the entire volume of the vaporized sample without reacting with it.

Thermo Scientific Chromatography Data Systems include the **Vapor Volume Calculator** that rapidly calculates the expansion volume of several factors (solvent, injected liquid volume, temperature, and inlet pressure) to help you determine if a liner dimension is suitable for a method.

#### **Split Liners**

Select an appropriate liner from Table 12.

 Table 12.
 Split Liners

No	Liner	Description
1		Deactivated liner 4-mm ID; Glass Wool; 900 μL theoretical volume.
2		Deactivated empty liner; 4-mm ID.
3		Mini Lam Deactivated empty liner; 4-mm ID.

#### **Splitless Liners**

Select an appropriate liner from Table 13.

 Table 13.
 Splitless Liners

No	Liner	Description
1		Deactivated Single Tapered Glass Wool; 900 µL theoretical volume.
2	4	Deactivated Single Tapered empty liner.

## **O-rings**

The internal (carrier line) and external (purge line) o-rings of the body head must be replaced if there are leaks. To replace the o-rings, please refer to *TRACE 1300/TRACE 1310 Hardware Manual*.

# **Using Instant Connect Helium Saver Injector Module Parameters**

This menu includes the operating parameters for the Instant Connect Helium Saver Injector Module. Editable parameters vary with the operating mode: split, splitless, surged splitless, and the flow mode: constant flow, constant pressure, programmed flow, programmed pressure. Using Instant Connect Helium Saver Injector Module Parameters

- Carrier Gas Parameters
- Injection Mode
- Inlet Parameters
- Purge Parameters
- Setting Gas Parameters
- Setting Parameters for the Split Mode
- Setting Parameters for the Splitless Mode
- Setting Parameters for the Surged Splitless Mode

The following sections list and describe the parameters controlling the front/back SSL injector. They are:

#### **Carrier Gas Parameters**

Set the carrier gas control parameters. Visualized parameters change according to the **Flow Mode** set.

**Pressure** — Defines the actual and setpoint pressure of the carrier gas. The range is On/Off; 5–1000 kPa (0.725-145 psi; 0.05-10 bar). This line is not editable when the Constant Flow or Programmed Flow mode is selected.

**Column Flow** — Defines the carrier gas flow rate through the column. The range is On/Off; 0.01-100 mL/min. Select **On** to display the actual and setpoint values. Select **Off** or **0** to turn off all inlet flows. This line is not editable when the Constant Pressure or Programmed Pressure mode is selected.

**Flow Mode** — Defines the carrier gas control mode to use. Each mode activates or deactivates the dedicated parameters.

- **Constant Flow** The column flow is kept constant throughout the analysis. The pressure at the column head will change with the column temperature to maintain a consistent flow.
- **Constant Pressure** The pressure at the column head is kept constant throughout the analysis. During a temperature program, the column flow decreases due to the increase of the carrier gas viscosity.
- **Programmed Flow** The column flow rate can be programmed to change during the analytical run for up to three flow ramps.

The parameters are:

- **Initial Flow** Defines the beginning flow rate.
- Initial Time Defines how long the Initial Flow is maintained.

- Ramp 1 The ramp rate in mL/min<sup>2</sup> to reach the final flow rate. Select On to enable the ramp and display the setpoint value.
- Final Flow The final flow rate the carrier gas will reach at the end of the ramp rate.
- **Final Time** Defines how long the corresponding Final flow must be maintained.
- Ramp 2-3 To program additional ramps, select On and enter the ramp rates in mL/min<sup>2</sup>. The Final Flow and Final Time menu items for the ramp are displayed. The ranges and functions of these menu items are identical to the Final Flow and Final Time menu items for Ramp 1.
- **Programmed Pressure** The inlet pressure can be programmed to change during the analytical run up to three pressure ramps.

The parameters are:

- Initial Pressure Defines the initial pressure.
- Initial Time Defines how long the Initial Pressure is maintained.
- Ramp 1 Defines the ramp pressure in kPa/min to reach the Final Pressure. Select
   On to enable the ramp and display the setpoint value.
- Final Pressure Defines the final pressure the carrier gas will reach at the end of the ramp rate.
- Final Time Defines how long the corresponding final pressure must be maintained.
- Ramp 2-3 To program additional ramps, select On and enter the ramp rates in kPa/min. The Final Pressure and Final Time menu items for the ramp are displayed. The ranges and functions of these menu items are identical to the Final Pressure and Final Time menu items for Ramp 1.

**Linear Velocity** — The calculated velocity of the carrier gas through the column, expressed in cm/s. It is not editable.

**Void Time** — The elution time of an un-retained peak, expressed in seconds. It is not editable.

**Gas Saver** — This function reduces the consumption of nitrogen pressurization gas. The range is On/Off; 5–500 mL/min. Select **On** to turn on the gas saver flow and display the setpoint values. Select **Off** to turn off the gas saver flow. The flow is retained in memory.

**Gas Saver Time** — Defines the time in the run when the gas saver function starts to operate. Usually it starts after the injection to conserve gas. Set a value from 0.00 to 999.99 min. This line does not appear if Gas saver flow is Off.

**Vacuum Comp**. — Use this parameter only when the TRACE 1300/TRACE 1310 GC is coupled with a mass spectrometer detector to compensate for vacuum column outlet. The range is On/Off.

# **Injection Mode**

Choose which injection mode to use with the SSL injector. Each mode activates or deactivates the dedicated parameters.

**Split** — The carrier flow is split in the injection port with the bulk going out the split vent. The split vent remains open all the time.

**Splitless** — Closes the split vent during injection to drive the entire sample into the column. Splitless times of about 1 minute are typical.

**Surged Splitless** — Same as **Splitless** but can also program a surge during an injection. Surge starts at Prep Run and continues until the surge duration time is finished. Surge is further defined in next group of controls.

#### **Inlet Parameters**

The SSL injection parameters are described below:

**Temperature** — Defines the setpoint for the injector's temperature. Depending on the injection mode you select, set a temperature high enough to vaporize the sample and the solvent. Enter a value for the inlet temperature in the range of 0-400 °C.

**Split Flow** — Enter a value in the range of 5-1250 mL/min. The **Split Ratio** is adjusted automatically. In addition, this value is governed by the initial column flow rate entered on the associated carrier gas control. If the flow rate is changed, the Split Flow value is adjusted so that the Split Ratio is maintained. However, if the Split Flow value then falls outside its limits, a warning is generated.

**Split Ratio** — Enabled when the injection mode is set to **Split**. It is also associated to the Flow Mode set to either Constant Flow or Programmed Flow. Specify the ratio of split flow to column flow. Calculate the split ratio: Split Ratio = (split flow) / (column flow). Enter a value in the range of 1-12500. The Split Flow entry is adjusted automatically.

**Splitless Time** — Enabled when the injection mode is set to either **Splitless** or **Surged Splitless**. Specify the length of time the split valve remains closed after a splitless injection. Enter a value in the range of 0.00-999.99 min. The timer begins at the start of the run. The split vent reopens when the splitless time ends.

#### **Purge Parameters**

Purge parameters are available when the injection mode is set to either **Splitless** or **Surged Splitless**.

**Constant Septum Purge** — Controls the septum purge for the injector. The choices are **On** or **Off**. Select **On** to activate the function to continuously flush the septum with a purge flow.

**Note** The purge flow, in the range of 0.5-50 mL/min, must be set in the Configuration Page of the injector module.

**Stop Purge For** — Enabled if the Constant Septum Purge parameter is set to **On**. You can then enter a time from 0.00 to 999.99 min at which the septum purge ceases.

#### **Surge Parameters**

Surge parameters are enabled when the injection mode is set to Surged Splitless.

**Surge Pressure** — Defines the pressure applied during the splitless time to produce a surge of flow in the injector to speed the transfer of the sample. Enter a value in the range of 5-1000 kPa (0.725-145 psi; 0.05-10 bar).

**Surge Duration** — Defines the time that the surge pressure is maintained. Enter a value in the range of 0.00-999.99 min. Typically, set to coincide with the **Splitless time**.

#### **Setting Gas Parameters**

Before starting, check that the carrier gas type is correct for the analysis.

#### ✤ To set gas parameters

- 1. Program the carrier gas flow.
  - a. Select Flow Mode.
  - b. Choose the mode you want from Constant flow, Constant Pressure, Programmed Flow or Programmed Pressure.
  - c. Enter the initial **Flow**, or **Pressure**.
    - i. If you select the **Constant Flow** mode, enter the desired **Column Flow** value. The necessary pressure is calculated and adjusted to maintain the constant flow.
    - ii. If you select **Constant Pressure** mode, enter the desired **Pressure** value.
  - d. Enter a **Programmed Flow/Programmed Pressure**.

**Note** When you select Programmed Flow or Programmed Pressure mode, the menu for the carrier contains parameters for up to three program ramps.

- i. Select **Progr Flow/Pressure**, scroll to Initial Flow/Press and enter the desired value. Press Enter.
- ii. Scroll to Initial Time and enter a value. This parameter ends the initial part of the program.
- e. Program the **Ramps**.
  - i. Scroll to Ramp 1 and enter the value.

- ii. Scroll to Final Flow 1/Pressure 1 and enter the final value for the ramp.
- iii. Scroll to Final Time 1 and enter the final time for Ramp 1. This operation ends the first ramp setting.
- iv. If you do not want a second ramp, leave Ramp 2 set to **Off**. To enter a second ramp, scroll to Ramp 2 and enter the value.
- v. Scroll to Final Flow 2/Pressure 2 and enter the final value for the ramp.
- vi. Scroll to Final Time 2 and enter the final time for Ramp 2. This operation ends the second ramp setting.
- vii. If you do not want a third ramp, leave Ramp 3 set to **Off**. To enter a third ramp, scroll to Ramp 3 and enter the value.
- viii. Scroll to Final Flow 3/Pressure 3 and enter the final value for the ramp.
- ix. Scroll to Final Time 3 and enter the final time for Ramp 3. This operation ends the third ramp setting.
- 2. If the TRACE 1300/TRACE 1310 GC is working with a mass spectrometer detector, set **Vacuum Compensation** to **On** to compensate for vacuum column outlet.

#### **Setting Parameters for the Split Mode**

Before starting, verify that the correct liner is installed into the injector body and the system is free of leak.

#### To set a Split injection

- 1. Program the carrier gas flow.
- 2. From the mode list, choose Split.
- 3. Set the injector Temperature.
- 4. If carrier **Flow** mode (Programmed or Constant) is selected, specify the **Split Flow** or **Split Ratio**.
  - a. If you want a specific **Split Flow**, enter that value. **Split Ratio** will be calculated.
  - b. If you want a specific **Split Ratio**, enter that value. **Split Flow** will be calculated.
- 5. If desired, turn on Gas Saver and set Gas Saver Time after the injection time.

#### **Setting Parameters for the Splitless Mode**

Before starting, verify that the correct liner is installed into the injector body and the system is free of leaks.

#### To set a Splitless injection

1. Program the carrier gas flow.

- 2. From the mode list, choose **Splitless**.
- 3. Set the injector **Temperature**.
- 4. Enter the **Splitless Time**.
- 5. If desired turn on **Constant Septum Purge** and enter into **Stop Purge For** how many minutes elapse before restarting the purge.
- 6. If desired, turn on Gas Saver and set Gas Saver Time after the injection time.

## Setting Parameters for the Surged Splitless Mode

Before starting, verify that the correct liner is installed into the injector body and the system is free of leaks.

#### ✤ To set a Surged Splitless injection

- 1. Program the carrier gas flow.
- 2. From the Mode list, choose Surged Splitless.
- 3. Set the injector Temperature.
- 4. Enter the **Splitless Time**.
- 5. If desired turn on **Constant Septum Purge** and enter into **Stop Purge For** how many minutes elapse before restarting the purge.
- 6. Set the values for Surge Pressure and Surge Duration.
- 7. If desired, turn on Gas Saver and set Gas Saver Time after the injection time.

# **Programmable Temperature Vaporizing Injector** (**PTV**) **Module**

This chapter describes the Programmable Temperature Vaporizing Injector module and provides instructions for programming the injector parameters.

#### Contents

- Module Overview
- Injection Techniques
- Consumables
- Using PTV Parameters

# **Module Overview**

The Programmable Temperature Vaporizing (PTV) injector module includes injector body, heater body, cooling fan, split and purge vent valves, filters, and digital pneumatics for the control of the carrier gas, and fittings for the connection to the analytical column.



Figure 107. Programmable Temperature Vaporizing Injector Module

The module and injector components are shown in Figure 108 and Figure 109.

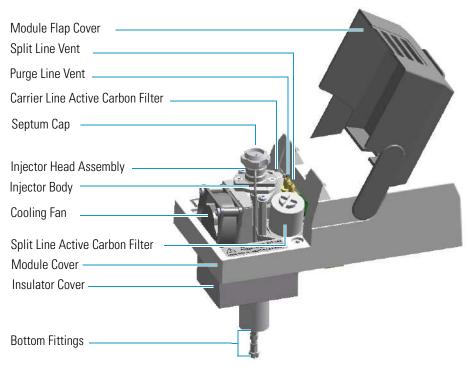
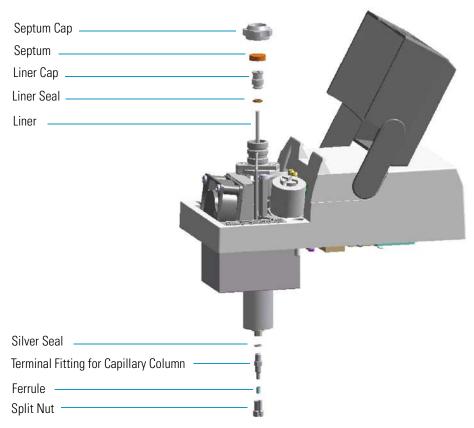


Figure 108. Programmable Temperature Vaporizing Injector Modules Components

Figure 109. Programmable Temperature Vaporizing Injector Components



The injector works at constant or programmed temperature into the range from ambient temperature up to 450 °C. Up to three temperature rates and plateaus are available when using the programmed temperature mode. The injection temperature is restored by a cooling fan located in the module.

Liquid nitrogen or carbon dioxide is used as a coolant for operating below ambient temperature (down to -100 °C with liquid nitrogen; down to -50 °C with carbon dioxide). The coolant flow is controlled by an optional cryogenic system.

PTV cryogenic systems with liquid nitrogen and with carbon dioxide are shown in Figure 110 and Figure 111 respectively.

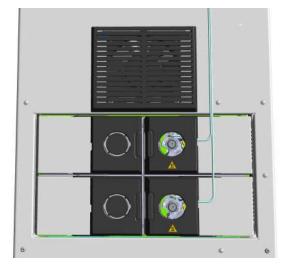






Figure 111. Front and Back PTV Cryo System with Carbon Dioxide





# **Injection Techniques**

There are three programmed temperature (PTV) operating modes: PTV Split, PTV Splitless, and PTV Large Volume; three constant temperature (CT) operating modes: CT Split, CT Splitless, and CT Surged Splitless; and an operating mode to use the PTV injector as an On-Column injector. You can configure and choose the operating mode from the injector parameter list.

# **PTV Split Mode**

In this mode the sample is injected into the liner in cold condition by keeping the initial temperature lower than the solvent boiling point. The sample is heated to the programmed vaporizing temperature and transferred into the capillary column. The final temperature should be suitable for vaporizing the component with the highest boiling point. During split injection the split valve is open. Only a part of the sample transfers into the column. The rest discharges through the split line. The split ratio of the split flow to the column flow determines the amount of sample that enters the chromatographic column.

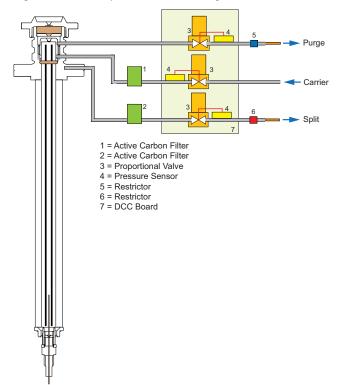


Figure 112. PTV Split Mode Block Diagram

Figure 113 shows an example of a temperature profile and timing of the valves in Split mode.

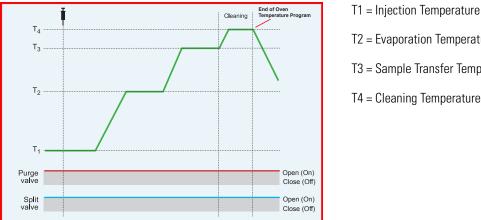


Figure 113. PTV Split Mode: Example of Temperature Profile and Timing

# T2 = Evaporation Temperature

- T3 = Sample Transfer Temperature
- T4 = Cleaning Temperature

# **PTV Splitless Mode**

In this mode the sample is injected into the liner in cold condition by keeping the initial temperature lower than the solvent boiling point. The sample is heated to the programmed vaporizing temperature and transferred into the capillary column. The final temperature should be suitable for vaporizing the component with the highest boiling point.

The splitless injection allows the entire sample to enter the column. The split line is closed during the sample injection and transfer to the column. Once the transfer is over, the split line reopens to flush the vaporization chamber of any remaining sample vapors. The time required to transfer the vaporized sample from the injector to the column is the **splitless time**. At the end of the splitless period, the split valve reopens, and the split flow flushes the injector of any remaining sample vapors.

A constant septum purge flow can continuously flush the septum throughout the analysis to reduce contamination from sample analytes.

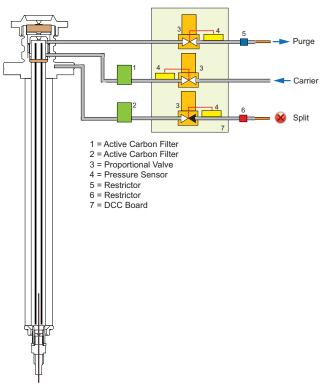
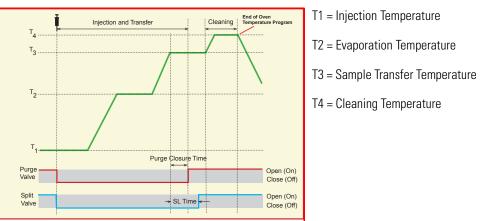


Figure 114. PTV Splitless Mode Block Diagram

Figure 115 shows an example of a temperature profile and timing of the valves in PTV Splitless mode.





## **PTV Large Volume Mode**

This mode eliminates the solvent before the sample enters the column. This allows large volume injections to increase the sensitivity of the analysis as well for normal injection volumes if the solvent or derivatizing reagents must be vented.

The sample is injected into the liner in cold condition. If injection parameters are properly set, analytes will stay in the liner while the solvent evaporates and is swept out.

Large volume injection is done in the following modes by using an autosampler:

1. **Speed Controlled Injection** — This mode is normally used for the single injection of large sample volumes (100-250  $\mu$ l). It is not intended for analysis of compounds with a boiling point close to that of the solvent.

The sample is injected at a controlled slow speed according to the temperature, the pressure, and the split flow in use so that during the injection a part of the solvent is eliminated through the split exit.

The evaporation speed is influenced by the temperature and flow and also by the type of packing present in the liner.

2. Multiple Injections — Perform multiple injections to increase the amount of compounds deposited in the liner. A small volume of sample is introduced several times with a delay between the injections. The volume of each injection is about 5-10  $\mu$ L. The initial analytical conditions must be maintained throughout the injections.

Figure 116 shows an example of a temperature profile and timing of the valves in Large Volume mode.

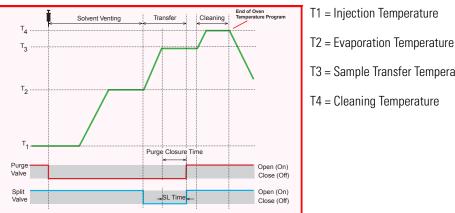


Figure 116. PTV Large Volume Mode: Example of Temperature Profile and Timing

T3 = Sample Transfer Temperature

T4 = Cleaning Temperature

#### **Constant Temperature Split Mode**

In this mode the sample is injected into the liner in hot condition by keeping the injection temperature suitable to the volatilities of the compounds.

During split injection the split and purge valves remain open throughout the run. Only a part of the sample transfers into the column. The rest discharges through the split line. The ratio of the split flow to the column flow (the **split ratio**) determines the amount of sample that enters the chromatographic column.

The PTV injector has a limited capacity due to the small volume of the liner used. In hot condition, the injection volume should not exceeding 1  $\mu$ L to prevent analytical problems caused by inlet overflow.

Figure 117 shows an example of a temperature profile and valves timing.

Figure 117. CT Split Mode Valves Timing



# **Constant Temperature Splitless Mode**

In this mode the sample is injected into the liner in hot condition by keeping the injection temperature suitable to the volatilities of the compounds.

The splitless injection allows the entire sample to enter the column. The split line is closed during the sample injection and transferred to the column. Once the transfer is over, the split line reopens to flush the vaporization chamber of any remaining sample vapors. The time required to transfer the vaporized sample from the injector to the column is the **splitless time**. At the end of the splitless period, the split valve reopens and the split flow flushes the injector of any remaining sample vapors.

A constant septum purge flow can continuously flush the septum throughout the analysis to reduce contamination from sample analytes.

Figure 118 shows an example of a temperature profile and valves timing.

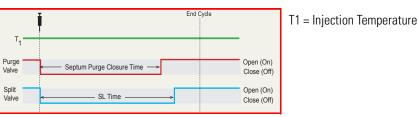


Figure 118. CT Splitless Mode Valves Timing

# **Constant Temperature Surged Splitless Mode**

In this mode the sample is injected into the liner in hot condition by keeping the injection temperature suitable to the volatilities of the compounds.

A carrier gas pressure surge activates during the injection phase for a preset time. The pressure applied during the splitless time produces a surge of flow in the injector, and accelerates the transfer process of the sample substances from the injector to the column.

This avoids the broadening of the sample band and the risk of the injector overloading.

A constant septum purge flow can continuously flush the septum throughout the analysis to reduce contamination from the sample's analytes.

#### **PTV On-Column Mode**

The PTV injector can be used similarly to an on-column injector if equipped with a special liner which has a restriction on top. See "Like On-Column Liners" on page 203. The restriction functions as a 0.47 mm OD needle guide, allowing you to inject a sample directly into a wide-bore column or a pre-column, by keeping the injector temperature lower than the solvent boiling point.

The injector heats with an automatic temperature rate emulating the oven temperature. When using this technique, set the initial oven temperature below the solvent boiling point.

**Note** This system can be used also with the AI 1310 autosampler setting the parameter **Post Dwell Time** to 10 seconds.

# Consumables

The consumables required for this injector are the septum and the glass liner.

#### Septum

You should always use good quality septa, such as the BTO septum supplied with the TRACE 1300/TRACE 1310. Such septa resist deformation, have longer life expectancy, and have a low bleed level, even at high temperatures.

PTV injector is compatible with the **Merlin Microseal<sup>™</sup> High Pressure Valve** instead of the septum.

## Liners

The sample injection mode used determines the choice of liner to install into the injector body. An appropriate liner must ensure complete sample vaporization and contain the entire volume of the vaporized sample without reacting with it.

Thermo Scientific Chromatography Data Systems include the **Vapor Volume Calculator** that rapidly calculates the expansion volume of several factors (solvent, injected liquid volume, temperature, and inlet pressure) to help you determine if a liner dimension is suitable for a method.

#### **Split Liners**

Select an appropriate liner from Table 14.

#### Table 14. Split Liners

No	Liner	Description
1		Silcosteel deactivated liner. 2mm ID; 2.75 mm OD; 120 mm length; 0.38 mL theoretical volume.
2		Non-deactivated liner. 2 mm ID; 2.75 mm OD; 120 mm length; 0.38 mL theoretical volume.
3		Deactivated glass liner. 2 mm ID; 2.75 mm OD; 120 mm length; 0.340 mL theoretical volume. The deactivated glass surface offers the advantage of a chemically inert environmental for injection of polar compounds.
4		Deactivated liner with a plug of silica wool. 2mm ID; 2.75 mm OD; 120 mm length; with silica wool. Very suitable for PTV Split and Backflush operations.

#### **Splitless Liners**

Select an appropriate liner from Table 15.

#### Table 15. Splitless Liners

No	Liner	Description
1		Silcosteel deactivated liner. 2 mm ID; 2.75 mm OD; 120 mm length; 0.38 mL theoretical volume.
2		Non-deactivated liner. 2 mm ID; 2.75 mm OD; 120 mm length; 0.38 mL theoretical volume.
3		Silcosteel deactivated liner. 1 mm ID; 2.75 mm OD; 120 mm length; 0.0095 mL theoretical volume. Used for injection of samples with high molecular weight compounds.
4		Deactivated liner. 1 mm ID; 2.75 mm OD; 120 mm length; 0.0095 mL theoretical volume. Used for injection of samples with high molecular weight compounds.
5		Deactivated glass liner. 2 mm ID; 2.75 mm OD; 120 mm length; 0.340 mL theoretical volume. The deactivated glass surface offers the advantage of a chemically inert environmental for injection of polar compounds.

#### Large Volume Liners

Select an appropriate liner from Table 16.

#### Table 16. Large Volume Liners

No	Liner	Description
1	タル ひか うか うか うか うか しん	Silcosteel deactivated liner with deactivated silica wool. 2 mm ID; 2.75 mm OD; 120 mm length; 0.38 mL theoretical volume.
2		Glass sintered deactivated liner without quartz wool for injection of polar and labile compounds. 1.2 mm ID; 2.75 mm OD; 120 mm length; 0.35 mL theoretical volume. The sintered porous surface coating (0.25-0.5 mm) holds the liquid during the controlled speed large volume injection while offering a chemically inert surface.
3		Deactivated glass liner with three baffles. 1.2 mm ID; 2.75 mm OD; 120 mm length; 0.180 mL theoretical volume. The deactivated surface with baffles allows increasing the injectable volumes with the 1 mm ID glass liner. Baffles can also be used for holding small amount of silica wool.
4		Liner with baffles for analyses of pesticides.

#### Like On-Column Liners

Select an appropriate liner from Table 17.

	Table	17.	Like	On-column	Liner
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No	Liner	Description
1		Silcosteel deactivated liner with a 0.6 mm ID restriction. 1 mm ID; 2.75 mm OD; 120 mm length; not remarkable theoretical volume. Used when the PTV operates like an on-column injector. The use of wide-bore column or wide-bore precolumn is required.

# **Using PTV Parameters**

The following section lists and describes the parameters controlling the PTV injector.

- Carrier Gas Parameters
- Injection Mode
- Inlet Parameters
- Purge Parameters
- Surge Parameters
- Injection Phases Parameters
- Setting Gas Parameters
- Setting Gas Parameters
- Setting Parameters for PTV Split Mode
- Setting Parameters for PTV Splitless Mode
- Setting Parameters for PTV Large Volume Mode
- Programming Injection Parameters
- Setting Parameters for CT Split Mode
- Setting Parameters for CT Splitless Mode
- Setting Parameters for CT Surged Splitless Mode

## **Carrier Gas Parameters**

Set the carrier gas control parameters, regardless of the carrier gas. Visualized parameters change according to the **Flow Mode** set.

**Pressure** — Defines the actual and setpoint pressure of the carrier gas. The range is On/Off; 5–1000 kPa (0.725-145 psi; 0.05-10 bar). This line is not editable when the Constant Flow or Programmed Flow mode is selected.

**Column Flow** — Defines the carrier gas flow rate through the column. The range is On/Off; 0.01-100 mL/min. Select **On** to display the actual and setpoint values. Select **Off** or **0** to turn off all inlet flows. This line is not editable when the Constant Pressure or Programmed Pressure mode is selected.

**Flow Mode** — Defines the carrier gas control mode to use. Each mode activates or deactivates the dedicated parameters.

- **Constant Flow** The column flow is kept constant throughout the analysis. The pressure at the column head will change with the column temperature to maintain a consistent flow.
- **Constant Pressure** The pressure at the column head is kept constant throughout the analysis. During a temperature program, the column flow decreases due to the increase of the carrier gas viscosity.
- **Programmed Flow** The column flow rate can be programmed to change during the analytical run for up to three flow ramps.

The parameters are:

- **Initial Flow** Defines the beginning flow rate.
- Initial Time Defines how long the Initial Flow is maintained.
- Ramp 1 The ramp rate in mL/min<sup>2</sup> to reach the final flow rate. Select On to enable the ramp and display the setpoint value.
- **Final Flow** The final flow rate the carrier gas will reach at the end of the ramp rate.
- **Final Time** Defines how long the corresponding Final flow must be maintained.
- Ramp 2-3 To program additional ramps, select On and enter the ramp rates in mL/min<sup>2</sup>. The Final Flow and Final Time menu items for the ramp are displayed. The ranges and functions of these menu items are identical to the Final Flow and Final Time menu items for Ramp 1.
- **Programmed Pressure** The inlet pressure can be programmed to change during the analytical run up to three pressure ramps.

The parameters are:

- Initial Pressure Defines the initial pressure.
- Initial Time Defines how long the Initial Pressure is maintained.
- Ramp 1 Defines the ramp pressure in kPa/min to reach the Final Pressure. Select
   On to enable the ramp and display the setpoint value.
- Final Pressure Defines the final pressure the carrier gas will reach at the end of the ramp rate.
- Final Time Defines how long the corresponding final pressure must be maintained.
- Ramp 2-3 To program additional ramps, select On and enter the ramp rates in kPa/min. The Final Pressure and Final Time menu items for the ramp are displayed. The ranges and functions of these menu items are identical to the Final Pressure and Final Time menu items for Ramp 1.

**Linear Velocity** — The calculated velocity of the carrier gas through the column, expressed in cm/s. It is not editable.

**Void Time** — The elution time of an un-retained peak, expressed in seconds. It is not editable.

**Gas Saver** — This function reduces carrier gas consumption. The range is On/Off; 5–500 mL/min. Select **On** to turn on the gas saver flow and display the setpoint values. Select **Off** to turn off the gas saver flow. The flow is retained in memory.

**Gas Saver Time** — Defines the time in the run when the gas saver function starts to operate. Usually it starts after the injection to conserve gas. Set a value from 0.00 to 999.99 min. This line does not appear if Gas saver flow is Off.

**Vacuum Comp.** — Use this parameter only when the TRACE 1300/TRACE 1310 is coupled with a mass detector to compensate for vacuum column outlet. The range is On/Off.

## **Injection Mode**

Choose which injection mode to use with the PTV injector. Each mode activates or deactivates the dedicated parameters.

**PTV Split** — Splits the carrier flow in the injection port with the bulk going out the split vent. The split vent remains open all the time.

**PTV Splitless** — Closes the split vent during injection to drive the entire sample into the column. Splitless times of about 1minute are typical.

PTV Large Volume — Eliminates the solvent before the sample enters the column.

**CT Split** — Performs traditional isothermal split operation. The carrier flow is split in the injection port with the bulk going out the split vent. The split vent remains open all the time.

**CT Splitless** — Performs traditional isothermal splitless operation. The split vent is closed during injection to drive the entire sample into the column. Splitless times of about 1 minute are typical.

**CT Surged Splitless** — Same as **Splitless** but can also program a surge during an injection. Surge starts at Prep Run and continues until the surge duration time is finished. Surge is further defined in next group of controls.

**On-Column** — Uses the PTV injector as an On-Column injector. The injector heats with an automatic temperature rate emulating the oven temperature. When using this technique, set the initial oven temperature below the solvent boiling point.

#### **Inlet Parameters**

The PTV injection parameters are described below.

**Temperature** — Defines the setpoint for the injector temperature. Depending on the injection mode you select, set the temperature high enough to vaporize the sample and solvent. Enter a value for the inlet temperature from 0 °C to 450 °C.

However, if you selected the Sub-ambient option, then you can enter cryogenic (below 0 °C) temperature values in the range from -50 °C to 450 °C with carbon dioxide, and from -100 °C to 450 °C with liquid nitrogen.

**Split Flow** — Enter a value from 5 to 1250 mL/min. The **Split Ratio** is adjusted automatically. In addition, this value is governed by the initial column flow rate entered on the associated carrier gas control. If the flow rate is changed, the Split Flow value is adjusted so that the split ratio is maintained. However, if the Split Flow value then falls outside its limits, a warning is generated.

**Split Ratio** — Enabled when the injection mode is set to either **Split** or **CT Split**. It is also associated the Flow Mode and set to either Constant Flow or Programmed Flow. Specify the ratio of Split Flow to column flow. Calculate the Split Ratio: Split Ratio = (Split Flow) / (Column Flow). Enter a value from 1 to 12500. The Split Flow entry is adjusted automatically.

**Splitless Time** — Enabled when the injection mode is set to either **Splitless, CT Splitless** or **CT Surged Splitless**. Specify the length of time the split valve remains closed after a splitless injection. Enter a value from 0.00 to 999.99 min. The timer begins at the start of the run. The split vent reopens when the splitless time ends.

#### **Purge Parameters**

Purge parameters are available when the injection Mode is set to **Splitless**, **Surged Splitless**, **CT Splitless**, or **CT Surged Splitless**.

**Constant Septum Purge** — Controls the septum purge for the injector. The range is On; Off. Select **On** to activate the function to continuously flush the septum with a purge flow.

**Note** The purge flow, in the range of 0.5-50 mL/min, must be set in the Configuration Page of the injector module.

**Stop Purge For** — Enabled if the Constant Septum Purge parameter is set to On. You can then enter a time from 0.00 to 999.99 min at which the septum purge ceases.

#### **Surge Parameters**

Surge parameters are enabled when the injection mode is set to either **Surged Splitless** or **CT Surged Splitless**.

**Surge Pressure** — Defines the pressure applied during the splitless time to produce a surge of flow in the injector to speed transfer of the sample. Enter a value from 5 to 1000 kPa.

**Surge Duration** — Defines the time for which the surge pressure is maintained. Enter a value from 0.00 to 999.99 min. Typically, set to coincide with the **Splitless time**.

### **Injection Phases Parameters**

There are four phases: Injection, Evaporation, Transfer, and Cleaning.

**Injection** — Specifies the inlet parameters during the injection phase when the injection mode is set to either **Splitless** or **Large Volume**.

The parameters are:

- **Inject Temperature** Defines the injector temperature during the injection. Enter a value in the range 0-450 °C; when cryo is enabled, the range is from -50 to 450 with carbon dioxide, and from -100 to 450 °C with liquid nitrogen.
- **Inject Time** Defines the time to maintain the temperature during and after the injection. Enter a value in the range 0.00–999.99 min.
- **Inject Pressure** Defines the pressure value during the injection phase. The range is On; Off; 5-1000 kPa (0.725-145 psi; 0.05-10 bar).
- **Injection Flow** Defines the split flow during the injection time. It discharges the solvent or the non-retained compounds during the large volume phase. The flow setpoint must be compatible with the available pressure set. Enter a value in the range of 5-1250 mL/min.

**Evaporation** — Specifies the solvent evaporation parameters when the **Evaporation** option has been configured.

The parameters are:

- **Evaporation Ramp** Defines the ramp rate to reach the programmed solvent evaporation temperature. Enter a value in the range of 0.1–14.5 °C/s in 0.1 °C/s increments.
- **Evaporation Temp.** Defines the solvent evaporation temperature. Enter a value in the range 0-450 °C; when cryo is enabled, the range is from -50 to 450 with carbon dioxide, and from -100 to 450 °C with liquid nitrogen.
- **Evaporation Time** Defines the time the programmed solvent evaporation temperature must be maintained. Enter a value in the range of 0.00–999.99 min.
- **Evaporation Pressure** Defines the pressure used during the solvent evaporation phase. The pressure is applied at the beginning of the evaporation temperature ramp. The range is On; Off; 5-1000 kPa (0.725-145 psi; 0.05-10 bar).
- Evaporation Flow It is available only in the PTV Large Volume mode. Defines the split flow during the evaporation time. The value may be different than Split Flow. Enter a value in the range 5-1250 mL/min.

**Transfer** — Specifies the parameters for the sample transfer into the column. Configure **Ramped Pressure** option to activate **Transfer** and **Pressure** parameters.

The parameters are:

- **Transfer Ramp** Defines the rate of the temperature ramp to reach the temperature for the sample transfer. Enter a value in the range 0.1–14.5 °C/s in 0.1 °C/s increments.
- **Transfer Temp** Defines the temperature at which the sample transfers into the column. Enter a value in the range 0-450 °C; when cryo is enabled, the range is from -50 to 450 with carbon dioxide, and from -100 to 450 °C with liquid nitrogen.
- **Transfer Time** Defines the time the programmed sample transfer temperature must be maintained. Enter a value in the range of 0.00–999.99 min.
- **Transfer Delay Time** It is active only when Large Volume mode is set. It delays the temperature ramp start after evaporation phase. Enter a value in the range 0.00–999.99 min.
- **Transfer Pressure** Defines he pressure used during the sample transfer phase. This pressure is applied at the beginning of the transfer temperature ramp. The range is On; Off; 5-1000 kPa (0.725-145 psi; 0.05-10 bar).

**Cleaning** — Specifies the parameter for the injector cleaning when the **Cleaning Phase** option has been configured.

#### The parameters are:

- **Clean Ramp** Defines the ramp rate to reach the programmed injector cleaning temperature. Enter a value in the range of 0.1–14.5 °C/s in 0.1 °C/s increments.
- **Clean Temp** Defines the injector temperature during the cleaning phases. Enter a value in the range of 0-450 °C; when cryo is enabled, the range is from -50 to 450 with carbon dioxide, and from -100 to 450 °C with liquid nitrogen.
- **Clean Time** Defines the time the programmed sample transfer temperature must be maintained. Enter a value in the range of 0.00–999.99 min.
- **Clean Flow** Increases the flow during the cleaning phase. The clean flow setpoint must be compatible with the pressure set. Enter a value in the range of 5-1250 mL/min.

#### **Setting Gas Parameters**

Before starting, check that the carrier gas type is correct for the analysis.

#### To set gas parameters

- 1. Program the carrier gas flow.
  - a. Select Flow Mode.

- b. Choose the mode you want choosing among Constant flow, Constant Pressure, Programmed Flow or Programmed Pressure.
- c. Enter the initial Flow or Pressure.
  - i. If you select **Constant Flow** mode, enter the desired **Column Flow** value. The pressure necessary is calculated and adjusted to maintain the constant flow.
  - ii. If you select **Constant Pressure** mode, enter the desired **Pressure** value.
- d. Enter a **Programmed Flow/Programmed Pressure**.

**Note** When you select Programmed Flow or Programmed Pressure mode, the menu for the carrier contains parameters for up to three program ramps.

- i. Select **Progr Flow/Pressure**, scroll to Initial Flow/Press and enter the desired value. Press Enter.
- ii. Scroll to Initial Time and enter a value. This parameter ends the initial part of the program.
- e. Program the **Ramps**.
  - i. Scroll to Ramp 1 and enter the value.
  - ii. Scroll to Final Flow 1/Pressure 1 and enter the final value for the ramp.
  - iii. Scroll to Final Time 1 and enter the final time for Ramp 1. This operation ends the first ramp setting.
  - iv. If you do not want a second ramp, leave Ramp 2 set to **Off**. To enter a second ramp, scroll to Ramp 2 and enter the value.
  - v. Scroll to Final Flow 2/Pressure 2 and enter the final value for the ramp.
  - vi. Scroll to Final Time 2 and enter the final time for Ramp 2. This operation ends the second ramp setting.
  - vii. If you do not want a third ramp, leave Ramp 3 set to **Off**. To enter a third ramp, scroll to Ramp 3and enter the value.
  - viii. Scroll to Final Flow 3/Pressure 3 and enter the final value for the ramp.
  - ix. Scroll to Final Time 3 and enter the final time for Ramp 3. This operation ends the third ramp setting.
- 2. If the TRACE 1300/TRACE 1310 is working with a mass detector, set **Vacuum Compensation** to **On** to compensate for vacuum column outlet.

## **Setting Parameters for PTV Split Mode**

Use this procedure to setup a PTV Split mode injection. In this mode the split and purge valves remain open during an entire run.

Before stating, verify that the correct liner is installed into the injector body and the system is free of leak.

#### \* To set a PTV Split injection

- 1. Program the carrier gas flow.
- 2. From the Mode list, choose PTV Split.
- 3. Set the injector Temperature.
- 4. If carrier **Flow** Mode (Programmed or Constant) is selected, specify the **Split Flow** or **Split Ratio**.
  - a. If you want a specific **Split Flow**, enter that value. **Split Ratio** will be calculated for you.
  - b. If you want a specific **Split Ratio**, enter that value. **Split Flow** will be calculated for you.
- 5. If desired, turn on Gas Saver and set Gas Saver Time after the injection time.
- 6. From the Injection Phase list, select the various programmed phases required. If you want programming temperature ramps, please see "Programming Injection Parameters" on page 212 for details.

## **Setting Parameters for PTV Splitless Mode**

Use the following procedure to setup a PTV Splitless injection. In PTV splitless mode, the split and purge valves are closed during the **Prep Run** phase and remain closed up to the end of the transfer time programmed.

Before stating, verify that the correct liner is installed into the injector body and the system is free of leak.

#### To set a PTV Splitless injection

- 1. Program the carrier gas flow.
- 2. From the Mode list, choose **PTV Splitless**.
- 3. Set the injector Temperature.
- 4. Enter the desired **Split Flow** value.
- 5. Enter the **Splitless Time**.
- 6. If desired turn on **Constant Septum Purge** and set in **Stop Purge For** how many minutes elapse before restarting the purge.
- 7. If desired, turn on Gas Saver and set Gas Saver Time after the injection time.
- 8. From the Injection Phase list, select the various programmed phases required. If you want programming temperature ramps, please see "Programming Injection Parameters" on page 212 for details.

# Setting Parameters for PTV Large Volume Mode

Use the following procedure to setup a PTV Large Volume injection. In PTV Large Volume mode, the purge valve must be normally closed during the Prep Run phase, and remains closed after the end of the transfer ramp for the programmed time. The split valve is closed only at the end of the injection time and evaporation time, if programmed. It remains closed up to the end of the transfer time programmed.

Before starting, verify that the correct liner is installed into the injector body and the system is free of leaks.

#### To setup a PTV Large Volume injection

- 1. Program the carrier gas flow.
- 2. From the Mode list, choose PTV Large Volume.
- 3. Set the injector **Temperature**.
- 4. Enter the desired **Split Flow** value.
- 5. Enter the **Splitless Time**.
- 6. If desired, turn on **Constant Septum Purge** and set in **Stop Purge For** how many minutes will elapse before restarting the purge.
- 7. From the Injection Phase list, select the various programmed phases required. If you want to program temperature ramps, please see "Programming Injection Parameters" on page 212 for details.

#### **Programming Injection Parameters**

Use the following sequence to program temperature ramps when operating in PTV Split, PTV Splitless, PTV Large Volume mode. Be sure to program the other operating mode parameters before programming the temperature ramps.

#### **PTV Injection Cycle**

Figure 119 shows a generic temperature program of the PTV injection cycle.

# Injection cycle End of Oven Temperature Prog Solvent aporation Sample transfer Stand-by

Figure 119. Generic Temperature Profile

- T1 = Injection Temperature
- T2 = Evaporation Temperature
- T3 = Sample Transfer Temperature
- T4 = Cleaning Temperature

- 1. Open the Injection Phase menu.
- 2. Set Ramped Pressure? Yes to program ramped pressure.
- 3. Enter the **Inject Pressure** at the beginning of the temperature ramp.
- 4. Enter the **Inject Time** at which the injector temperature must be maintained.
- 5. Set the sample Transfer pressure.
- 6. Set the sample Transfer temperature.
- 7. Set the Transfer ramp rate in °C/s to reach the sample transfer temperature.
- 8. Set the Transfer time for which the transfer temperature must be maintained.

**IMPORTANT** Consider a time almost similar to the oven temperature program unless the cleaning phase is used.

9. Only in PTV Large Volume mode, set the Injection flow for the solvent venting phase.

#### If Solvent Evaporation Has Been Configured

- 1. Set the initial **Evaporation pressure** for the evaporation temperature ramp during the solvent evaporation phase.
- 2. Set the **Evaporation ramp** rate in °C/s to reach the solvent evaporation temperature.
- 3. Set the solvent **Evaporation temperature**.
- 4. Set the **Evaporation time** at which the transfer temperature must be maintained.
- 5. Set the **Evaporation flow** for the solvent venting in **PTV Large Volume** mode.

#### If Injector Cleaning Has Been Configured

- 1. Set the injector **Clean temp**.
- 2. Set the **Clean ramp** rate in °C/s to reach the cleaning temperature.
- 3. Set the **Clean time** at which the cleaning temperature must be maintained.
- 4. Set the Clean flow value to increase the flow during the cleaning phase.

**Note** Inject pressure, Transfer pressure, and Evaporation pressure parameters will not be displayed if Ramped pressure parameter is set to No. If Ramped pressure parameter is set to Yes in Inject Phase Menu, please read the considerations reported in Ramped Pressure Option in Menu Inject Phase Menu.

#### **Ramped Pressure Option in Menu Inject Phase Menu**

When ramped pressure is enabled during the injection phases, please consider the following:

- Independently of the flow mode selected, the pressure during the Injection, Evaporation and Transfer will be controlled through the PTV Menu. At the end of the Transfer Time on the PTV, control of the carrier will once again be managed through the Carrier Gas menu. This means that the pressure/flow will be back to the value defined in the Carrier Gas menu for that moment of the analysis. If programmed pressure or programmed flow mode has been selected, the pressure/flow program will virtually begin at the start of the oven temperature program, and then the pressure/flow will assume the value defined in the pressure/flow program for that moment of the analysis.
- If the temperature of the injector must be kept constant during the whole oven temperature program, it is preferable to use the cleaning phase instead of the transfer phase. This because during the cleaning the carrier control is managed as defined in the Carrier Gas Menu (whereas during the transfer phase the carrier control is managed as defined in the PTV control Menu).

## **Setting Parameters for CT Split Mode**

Use this procedure to setup a CT Split mode injection. In this mode, the split and purge valves remain open throughout run.

Before stating, verify that the correct liner is installed into the injector body and the system is free of leak.

#### To set a CT Split injection

- 1. Program the carrier gas flow.
- 2. From the Mode list, choose CT Split.
- 3. Set the injector Temperature.
- 4. If Flow Mode (Programmed or Constant) is selected, specify the Split Flow or Split Ratio.
  - a. If you want a specific **Split Flow**, enter that value. **Split Ratio** will be calculated for you.
  - b. If you want a specific **Split Ratio**, enter that value. **Split Flow** will be calculated for you.
- 5. If desired, turn on Gas Saver and set Gas Saver Time after the injection time.

## **Setting Parameters for CT Splitless Mode**

Use the following procedure to setup a CT Splitless injection. In this mode, the split and purge valves are closed during the Prep Run phase and remain closed after the injection for the programmed duration.

Before stating, verify that the correct liner is installed into the injector body and the system is free of leak.

#### \* To set a CT Splitless injection

- 1. Program the carrier gas flow.
- 2. From the Mode list, choose Splitless.
- 3. Set the injector Temperature.
- 4. Enter the **Splitless Time**.
- 5. If desired turn on **Constant Septum Purge** and set in **Stop Purge For** how many minutes elapse before restarting the purge.
- 6. If desired, turn on Gas Saver and set Gas Saver Time after the injection time.

### **Setting Parameters for CT Surged Splitless Mode**

Use the following procedure to setup a CT Surged Splitless injection. In this mode, a carrier gas pressure surge activates during the injection phase for a programmed time. Surge accelerates the transfer process of the substances from the injector to the column. The pressure pulse starts in the **Prep Run** phase and lasts until the end of the programmed surge duration. The split and purge valves close during the **Prep Run** phase and remain closed after injection for the programmed duration.

Before stating, verify that the correct liner is installed into the injector body and the system is free of leak.

#### ✤ To set a CT Surged Splitless injection

- 1. Program the carrier gas flow.
- 2. From the Mode list, choose CT Surged Splitless.
- 3. Set the injector Temperature.
- 4. Enter the **Splitless Time**.
- 5. If desired turn on **Constant Septum Purge** and set in **Stop Purge For** how many minutes elapse before restarting the purge.
- 6. Set the values for Surge Pressure and Surge Duration.
- 7. If desired, turn on Gas Saver and set Gas Saver Time after the injection time.

11

# **Programmable Temperature Vaporizing Backflush Injector (PTVBKF) Module**

This chapter describes the Programmable Temperature Vaporizing Injector module for **Backflush** applications and provides instructions for programming the injector parameters.

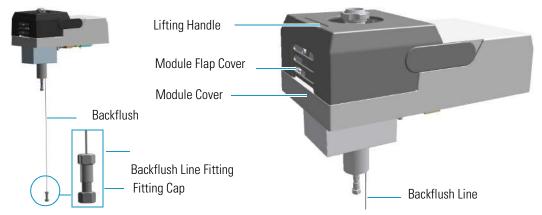
#### Contents

- Module Overview
- Backflush Mode
- Consumables
- Using PTVBKF Parameters
- Connecting PTV Backflush System

### **Module Overview**

The PTV Backflush module includes injector body, heater body, cooling fan, split and purge vent valves, filters, digital pneumatics for the control of the carrier gas, backflush control, backflush line, and fittings for the connection to the analytical column.

Figure 120. PTV Backflush Injector Module



The module and injector components are shown in Figure 121 and Figure 122.

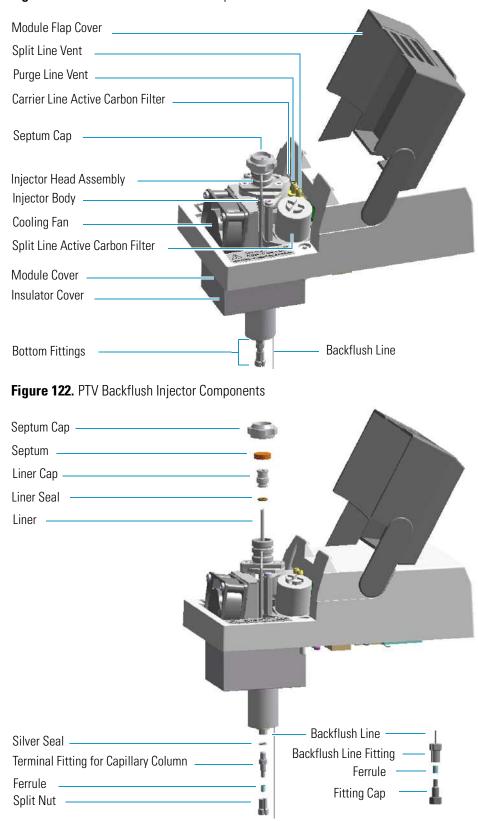


Figure 121. PTV Backflush Module Components

The injector works at constant or programmed temperature into the range from ambient temperature up to 450 °C.

Up to three temperature rates and plateaus are available when programmed temperature mode is used. The injection temperature is restored by a cooling fan located in the module.

Liquid nitrogen or carbon dioxide is used as a coolant for operating below ambient temperature (down to -100 °C with liquid nitrogen; down to -50 °C with carbon dioxide). The coolant flow is controlled by an optional cryogenic system.

PTVBKF cryo systems with liquid nitrogen and with carbon are shown in Figure 123 and Figure 124 respectively.

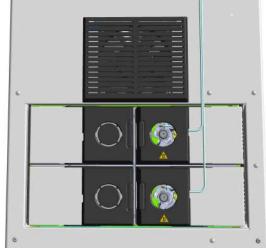




Figure 123. Front and Back PTV Cryo System with Liquid Nitrogen

Figure 124. Front and Back PTV Cryo System with Carbon Dioxide





# **Backflush Mode**

This operating mode allows elimination of the solvent before the sample enters the column and also, during the cleaning phase, the heavy part of the sample, which is not relevant for the analysis. This mode is suitable for Large Volume Injection and for sample introduction into a GC/Mass Spectrometer. The sample is injected into the liner in cold condition. If injection parameters are properly set, analytes will stay in the liner while the solvent evaporates and is swept out.

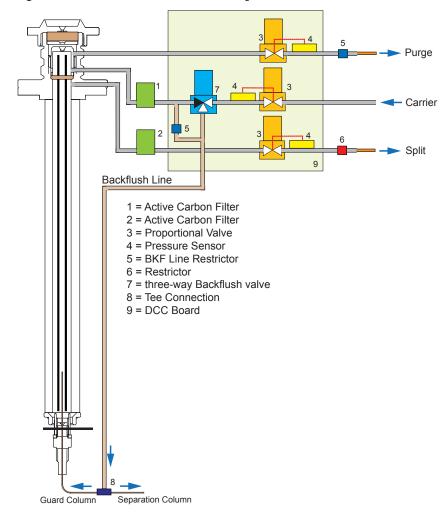


Figure 125. PTV Backflush Mode Block Diagram

Figure 126 shows an example of a temperature profile and timing of the valves in Backflush mode.

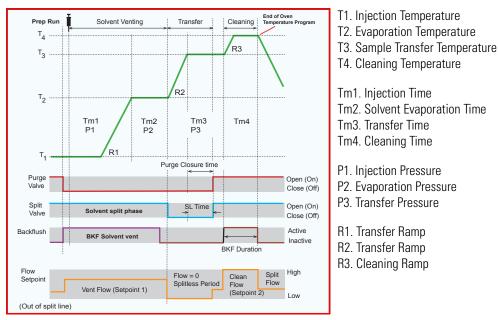


Figure 126. Temperature Profile and Timing in Backflush

**Note** The PTV Backflush module can be used in PTV Split, PTV Splitless, PTV Large Volume, CT Split, CT Splitless, and CT Surged Splitless modes after a proper modification of the backflush line. See Chapter 10, "Programmable Temperature Vaporizing Injector (PTV) Module."

# Consumables

The consumables required for this injector are the septum and the glass liner.

### Septum

You should always use good quality septa, such as the BTO septum supplied with the TRACE 1300/TRACE 1310. Such septa resist deformation, have longer life expectancy, and have a low bleed level, even at high temperatures.

PTV injector is compatible with the **Merlin Microseal<sup>™</sup> High Pressure Valve** instead of the septum.

### Liners

The sample injection mode used determines the choice of liner to install into the injector body. An appropriate liner must ensure complete sample vaporization and contain the entire volume of the vaporized sample without reacting with it. Thermo Scientific Chromatography Data Systems include the **Vapor Volume Calculator** that rapidly calculates the expansion volume of several factors (solvent, injected liquid volume, temperature, and inlet pressure) to help you determine if a liner dimension is suitable for a method.

### **Splitless Liners**

Select an appropriate liner from Table 18.

Table 18.	Splitless Liner	ſS
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No	Liner	Description
1		Silcosteel deactivated liner. 2mm ID; 2.75 mm OD; 120 mm length; 0.38 mL theoretical volume.
2		Non-deactivated liner. 2mm ID; 2.75 mm OD; 120 mm length; 0.38 mL theoretical volume.
3		Silcosteel deactivated liner. 1 mm ID; 2.75 mm OD; 120 mm length; 0.0095 mL theoretical volume. Used for injection of samples with high molecular weight compounds.
4		Deactivated liner. 1 mm ID; 2.75 mm OD; 120 mm length; 0.0095 mL theoretical volume. Used for injection of samples with high molecular weight compounds.
5		Deactivated glass liner. 2mm ID; 2.75 mm OD; 120 mm length; 0.340 mL theoretical volume. The deactivated glass surface offers the advantage of a chemically inert environmental for injection of polar compounds.

### **Large Volume Liners**

Select an appropriate liner from Table 19.

 Table 19.
 Large Volume Liners (Sheet 1 of 2)

No	Liner	Description
1	へた くを うを ふや ふた くん しん	Silcosteel deactivated liner with deactivated silica wool. 2 mm ID; 2.75 mm OD; 120 mm length; 0.38 mL theoretical volume.
2		Glass sintered deactivated liner without quartz wool for injection of polar and labile compounds. 1.2 mm ID; 2.75 mm OD; 120 mm length; 0.35 mL theoretical volume. Used instead of liner PN 45322056. The sintered porous surface coating (0.25-0.5 mm) holds the liquid during the controlled speed large volume injection while offering a chemically inert surface.

Table 19. Large Volume Liners (Sheet 2 of 2)

No	Liner	Description
3		Deactivated glass liner with three baffles. 1.2 mm ID; 2.75 mm OD; 120 mm length; 0.180 mL theoretical volume. The deactivated surface with baffles allows increasing the injectable volumes with the 1 mm ID glass liner. Baffles can also be used for holding small amount of silica wool.
4		Liner with baffles for analyses of pesticides.

## **Using PTVBKF Parameters**

The following section lists and describes the parameters controlling the PTVBKF injector.

- Carrier Gas Parameters
- Injection Mode
- Inlet Parameters
- Purge Parameters
- Surge Parameters
- Setting Gas Parameters
- Setting Gas Parameters
- Setting Parameters for Backflush
- Programming Injection Parameters

### **Carrier Gas Parameters**

Set the carrier gas control parameters, regardless of the carrier gas. Visualized parameters change according to the **Flow Mode** set.

**Pressure** — Defines the actual and setpoint pressure of the carrier gas. The range is On/Off; 5–1000 kPa (0.725-145 psi; 0.05-10 bar). This line is not editable when the Constant Flow or Programmed Flow mode is selected.

**Column Flow** — Defines the carrier gas flow rate through the column. The range is On/Off; 0.01-100 mL/min. Select **On** to display the actual and setpoint values. Select **Off** or **0** to turn off all inlet flows. This line is not editable when the Constant Pressure or Programmed Pressure mode is selected.

**Flow Mode** — Defines the carrier gas control mode to use. Each mode activates or deactivates the dedicated parameters.

- **Constant Flow** The column flow is kept constant throughout the analysis. The pressure at the column head will change with the column temperature to maintain a consistent flow.
- **Constant Pressure** The pressure at the column head is kept constant throughout the analysis. During a temperature program, the column flow decreases due to the increase of the carrier gas viscosity.
- **Programmed Flow** The column flow rate can be programmed to change during the analytical run for up to three flow ramps.

The parameters are:

- **Initial Flow** Defines the beginning flow rate.
- Initial Time Defines how long the Initial Flow is maintained.
- Ramp 1 The ramp rate in mL/min<sup>2</sup> to reach the final flow rate. Select On to enable the ramp and display the setpoint value.
- **Final Flow** The final flow rate the carrier gas will reach at the end of the ramp rate.
- **Final Time** Defines how long the corresponding Final flow must be maintained.
- Ramp 2-3 To program additional ramps, select On and enter the ramp rates in mL/min<sup>2</sup>. The Final Flow and Final Time menu items for the ramp are displayed. The ranges and functions of these menu items are identical to the Final Flow and Final Time menu items for Ramp 1.
- **Programmed Pressure** The inlet pressure can be programmed to change during the analytical run up to three pressure ramps.

The parameters are:

- Initial Pressure Defines the initial pressure.
- Initial Time Defines how long the Initial Pressure is maintained.
- Ramp 1 Defines the ramp pressure in kPa/min to reach the Final Pressure. Select
   On to enable the ramp and display the setpoint value.
- Final Pressure Defines the final pressure the carrier gas will reach at the end of the ramp rate.
- Final Time Defines how long the corresponding final pressure must be maintained.
- Ramp 2-3 To program additional ramps, select On and enter the ramp rates in kPa/min. The Final Pressure and Final Time menu items for the ramp are displayed. The ranges and functions of these menu items are identical to the Final Pressure and Final Time menu items for Ramp 1.

**Linear Velocity** — The calculated velocity of the carrier gas through the column, expressed in cm/s. It is not editable.

**Void Time** — The elution time of an un-retained peak, expressed in seconds. It is not editable.

**Gas Saver** — This function reduces carrier gas consumption. The range is On/Off; 5–500 mL/min. Select **On** to turn on the gas saver flow and display the setpoint values. Select **Off** to turn off the gas saver flow. The flow is retained in memory.

**Gas Saver Time** — Defines the time in the run when the gas saver function starts to operate. Usually it starts after the injection to conserve gas. Set a value from 0.00 to 999.99 min. This line does not appear if Gas saver flow is Off.

**Vacuum Comp**. — Use this parameter only when the TRACE 1300/TRACE 1310 is coupled with a mass detector to compensate for vacuum column outlet. The range is On/Off.

### **Injection Mode**

Choose which injection mode to use with the PTVBKF injector. Each mode activates or deactivates the dedicated parameters.

**PTV Split** — Splits the carrier flow in the injection port with the bulk going out the split vent. The split vent remains open all the time.

**PTV Splitless** — Closes the split vent during injection to drive the entire sample into the column. Splitless times of about 1 minute are typical.

PTV Large Volume — Eliminates the solvent before the sample enters the column.

**CT Split** — Performs traditional isothermal split operation. The carrier flow is split in the injection port with the bulk going out the split vent. The split vent remains open all the time.

**CT Splitless** — Performs traditional isothermal splitless operation. The split vent is closed during injection to drive the entire sample into the column. Splitless times of about 1 minute are typical.

**CT Surged Splitless** — Same as **Splitless** but can also program a surge during an injection. Surge starts at Prep Run and continues until the surge duration time is finished. Surge is further defined in next group of controls.

### **Inlet Parameters**

The PTVBKF injection parameters are described below.

**Temperature** — Defines the setpoint for the injector temperature. Depending on the injection mode you select, set the temperature high enough to vaporize the sample and solvent. Enter a value for the inlet temperature from 0 °C to 450 °C.

However, if you selected the Sub-ambient option, then you can enter cryogenic (below 0 °C) temperature values in the range from -50 °C to 450 °C with carbon dioxide, and from -100 °C to 450 °C with liquid nitrogen.

**Split Flow** — Enter a value from 5 to 1250 mL/min. The **Split Ratio** is adjusted automatically. In addition, this value is governed by the initial column flow rate entered on the associated carrier gas control. If the flow rate is changed, the Split Flow value is adjusted so that the split ratio is maintained. However, if the Split Flow value then falls outside its limits, a warning is generated.

**Split Ratio** — Enabled when the injection mode is set to either **Split** or **CT Split**. It is also associated the Flow Mode and set to either Constant Flow or Programmed Flow. Specify the ratio of Split Flow to column flow. Calculate the Split Ratio: Split Ratio = (Split Flow) / (Column Flow). Enter a value from 1 to 12500. The Split Flow entry is adjusted automatically.

**Splitless Time** — Enabled when the injection mode is set to either **Splitless, CT Splitless** or **CT Surged Splitless**. Specify the length of time the split valve remains closed after a splitless injection. Enter a value from 0.00 to 999.99 min. The timer begins at the start of the run. The split vent reopens when the splitless time ends.

### **Purge Parameters**

Purge parameters are available when the injection Mode is set to **Splitless**, **Surged Splitless**, **CT Splitless**, or **CT Surged Splitless**.

**Constant Septum Purge** — Controls the septum purge for the injector. The range is On; Off. Select **On** to activate the function to continuously flush the septum with a purge flow.

**Note** The purge flow, in the range of 0.5-50 mL/min, must be set in the Configuration Page of the injector module.

**Stop Purge For** — Enabled if the Constant Septum Purge parameter is set to On. You can then enter a time from 0.00 to 999.99 min at which the septum purge ceases.

### **Surge Parameters**

Surge parameters are enabled when the injection mode is set to either **Surged Splitless** or **CT Surged Splitless**.

**Surge Pressure** — Defines the pressure applied during the splitless time to produce a surge of flow in the injector to speed transfer of the sample. Enter a value from 5 to 1000 kPa (0.725-145 psi; 0.05-10 bar).

**Surge Duration** — Defines the time for which the surge pressure is maintained. Enter a value from 0.00 to 999.99 min. Typically, set to coincide with the **Splitless time**.

### **Injection Phases Parameters**

There are four phases: Injection, Evaporation, Transfer, and Cleaning.

**Injection** — Specifies the inlet parameters during the injection phase when the injection mode is set to either **Splitless** or **Large Volume**.

The parameters are:

- **Inject Temperature** Defines the injector temperature during the injection. Enter a value in the range of 0-450 °C; when cryo is enabled, the range is from -50 to 450 with carbon dioxide, and from -100 to 450 °C with liquid nitrogen.
- **Inject Time** Defines the time to maintain the temperature during and after the injection. Enter a value in the range of 0.00–999.99 min.
- **Inject Pressure** Defines the pressure value during the injection phase. The range is On; Off; 5-1000 kPa (0.725-145 psi; 0.05-10 bar).
- **Injection Flow** Defines the split flow during the injection phase. It discharges the solvent or the non-retained compounds during the large volume phase. The flow setpoint must be compatible with the available pressure set. Enter a value in the range of 5-1250 mL/min.

**Evaporation** — Specifies the solvent evaporation parameters when the **Evaporation** option has been configured.

The parameters are:

- **Evaporation Ramp** Defines the ramp rate to reach the programmed solvent evaporation temperature. Enter a value in the range of 0.1–14.5 °C/s in 0.1 °C/s increments.
- Evaporation Temp. Defines the solvent evaporation temperature. Enter a value in the range of 0-450 °C; when cryo is enabled, the range is from -50 to 450 with carbon dioxide, and from -100 to 450 °C with liquid nitrogen.
- **Evaporation Time** Defines the time the programmed solvent evaporation temperature must be maintained. Enter a value in the range of 0.00–999.99 min.
- **Evaporation Pressure** Defines the pressure used during the solvent evaporation phase. The pressure is applied at the beginning of the evaporation temperature ramp. The range is On; Off; 5-1000 kPa (0.725-145 psi; 0.05-10 bar).
- **Evaporation Flow** Defines the split flow during the evaporation time. The value may be different than Split Flow. Enter a value in the range of 5-1250 mL/min.

**Transfer** — Specifies the parameters for the sample transfer into the column only when the injection Mode is set to **Large Volume**. Configure **Ramped Pressure** option to activate **Transfer** and **Pressure** parameters.

The parameters are:

- **Transfer Ramp** Defines the rate of the temperature ramp to reach the temperature for the sample transfer. Enter a value in the range of 0.1–14.5 °C/s in 0.1 °C/s increments.
- **Transfer Temp** Defines the temperature at which the sample transfers into the column. Enter a value in the range of 0-450 °C; when cryo is enabled, the range is from -50 to 450 with carbon dioxide, and from -100 to 450 °C with liquid nitrogen.
- **Transfer Time** Defines the time the programmed sample transfer temperature must be maintained. Enter a value in the range of 0.00–999.99 min.
- **Transfer Delay Time** It is active only when the Large Volume mode is set. It delays the temperature ramp start after evaporation phase. Enter a value in the range of 0.00–999.99 min.
- **Transfer Pressure** Defines he pressure used during the sample transfer phase. This pressure is applied at the beginning of the transfer temperature ramp. The range is On; Off; 5-1000 kPa (0.725-145 psi; 0.05-10 bar).

**Cleaning** — Specifies the parameter for the injector cleaning when the **Cleaning Phase** option has been configured.

The parameters are:

- **Clean Ramp** Defines the ramp rate to reach the programmed injector cleaning temperature. Enter a value in the range of 0.1–14.5 °C/s in 0.1 °C/s increments.
- **Clean Temp** Defines the injector temperature during the cleaning phases. Enter a value in the range of 0-450 °C; when cryo is enabled, the range is from -50 to 450 with carbon dioxide, and from -100 to 450 °C with liquid nitrogen.
- **Clean Time** Defines he time the programmed sample transfer temperature must be maintained. Enter a value in the range of 0.00–999.99 min.
- **Clean Flow** Increases the flow during the cleaning phase. The clean flow setpoint must be compatible with the pressure set. Enter a value in the range of 5-1250 mL/min.

### **Setting Gas Parameters**

Before starting, check that the carrier gas type is correct for the analysis.

#### To set gas parameters

- 1. Program the carrier gas flow.
  - a. Select Flow Mode.
  - b. Choose the mode you want choosing among Constant flow, Constant Pressure, Programmed Flow or Programmed Pressure.
  - c. Enter the initial Flow or Pressure.
    - i. If you select **Constant Flow** mode, enter the desired **Column Flow** value. The necessary pressure is calculated and adjusted to maintain the constant flow.

- ii. If you select **Constant Pressure** mode, enter the desired **Pressure** value.
- d. Enter a **Programmed Flow/Programmed Pressure**.

**Note** When you select Programmed Flow or Programmed Pressure mode, the menu for the carrier contains parameters for up to three program ramps.

- i. Select **Progr Flow/Pressure**, scroll to Initial Flow/Press and enter the desired value. Press Enter.
- ii. Scroll to Initial Time and enter a value. This parameter ends the initial part of the program.
- e. Program the **Ramps**.
  - i. Scroll to Ramp 1 and enter the value.
  - ii. Scroll to Final Flow 1/Pressure 1 and enter the final value for the ramp.
  - iii. Scroll to Final Time 1 and enter the final time for Ramp 1. This operation ends the first ramp setting.
  - iv. If you do not want a second ramp, leave Ramp 2 set to **Off**. To enter a second ramp, scroll to Ramp 2 and enter the value.
  - v. Scroll to Final Flow 2 /Pressure 2 and enter the final value for the ramp.
  - vi. Scroll to Final Time 2 and enter the final time for Ramp 2. This operation ends the second ramp setting.
  - vii. If you do not want a third ramp, leave Ramp 3 set to **Off**. To enter a third ramp, scroll to Ramp 3and enter the value.
  - viii. Scroll to Final Flow 3/Pressure 3 and enter the final value for the ramp.
  - ix. Scroll to Final Time 3 and enter the final time for Ramp 3. This operation ends the third ramp setting.
- 2. If the TRACE 1300/TRACE 1310 is working with a mass detector, set **Vacuum Compensation** to **On** to compensate for vacuum column outlet.

### **Setting Parameters for Backflush**

Use the following procedure to setup a Backflush injection. The Backflush with PTV module can be activated in split and splitless mode only if the cleaning phase is enabled. For the activation of the Backflush in CT, see the section "Setting Parameters for Backflush" on page 162 in Chapter 7, "Split/Splitless Backflush Injector (SSLBKF) Module."

In **Large volume** mode the backflush can be activated in injection/evaporation phase and/or in cleaning phase if enabled. In this mode, the purge valve must be normally closed during the Prep Run phase, and remains closed after the end of the transfer ramp for the programmed time. The split valve is closed only at the end of the injection time and evaporation time, if programmed. It remains closed up to the end of the transfer time programmed.

Before starting verify that the correct liner is installed into the injector body and the system is free of leaks.

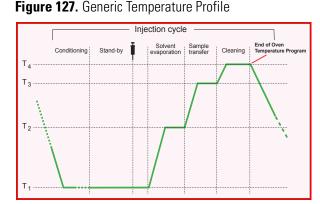
- 1. Program the carrier gas flow.
- 2. From the Mode list, choose PTV Large Volume.
- 3. Set the injector **Temperature**.
- 4. Enter the desired **Split Flow** value.
- 5. Enter the **Splitless Time**.
- 6. If desired turn on **Constant Septum Purge** and set in **Stop Purge For** how many minutes will elapse before restarting the purge.
- 7. From the Injection Phase list, select the various programmed phases required. If you want to program temperature ramps, please see "Programming Injection Parameters" on page 230 for details.

### **Programming Injection Parameters**

Use the following sequence to program temperature ramps when operating in PTV Split, PTV Splitless, PTV Large Volume mode. Be sure to program the other operating mode parameters before programming the temperature ramps.

#### **PTV Injection Cycle**

Figure 127 shows a generic temperature program of the PTV injection cycle.



T1 = Injection Temperature

- T2 = Evaporation Temperature
- T3 = Sample Transfer Temperature
- T4 = Cleaning Temperature

- 1. Open the Injection Phase menu.
- 2. Set **Ramped Pressure? Yes** to program ramped pressure.
- 3. Enter the **Inject Pressure** at the beginning of the temperature ramp.
- 4. Enter the **Inject Time** at which the injector temperature must be maintained.
- 5. Set the sample Transfer pressure.

- 6. Set the sample Transfer temperature.
- 7. Set the Transfer ramp rate in °C/s to reach the sample transfer temperature.
- 8. Set the **Transfer time** at which the transfer temperature must be maintained.

**IMPORTANT** Consider a time almost similar to the oven temperature program unless the cleaning phase is used.

9. Only in **PTV Large Volume** mode, set the **Injection flow** for the solvent venting phase.

#### If Solvent Evaporation Has Been Configured

- 1. Set the initial **Evaporation pressure** for the evaporation temperature ramp during the solvent evaporation phase.
- 2. Set the **Evaporation ramp** rate in °C/s to reach the solvent evaporation temperature.
- 3. Set the solvent Evaporation temperature.
- 4. Set the **Evaporation time** at which the transfer temperature must be maintained.

Set the Evaporation flow for the solvent venting in PTV Large Volume mode.

#### If Injector Cleaning Has Been Configured

- 1. Set the injector **Cleaning temp**.
- 2. Set the **Cleaning ramp** rate in °C/s to reach the cleaning temperature.
- 3. Set the **Cleaning time** at which the cleaning temperature must be maintained.
- 4. Set the **Clean flow** value to increase the flow during the cleaning phase.

**Note** Inject pressure, Transfer pressure, and Evaporation pressure parameters will not be displayed if Ramped pressure parameter is set to No. If Ramped pressure parameter is set to Yes in Inject Phase Menu, please read the considerations reported in Ramped Pressure Option in Menu Inject Phase Menu.

#### **Ramped Pressure Option in Menu Inject Phase Menu**

When ramped pressure is enabled during the injection phases, please consider the following:

• Independently of the flow mode selected, the pressure during the Injection, Evaporation and Transfer will be controlled through the PTV Menu. At the end of the Transfer Time on the PTV, control of the carrier once again be managed through the Carrier Gas menu. This means that the pressure/flow will be back to the value defined in the Carrier Gas menu for that moment of the analysis. If programmed pressure or programmed flow mode has been selected, the pressure/flow program will virtually begin at the start of the oven temperature program, then the pressure/flow will assume the value defined in the pressure/flow program for that moment of the analysis.

• If the temperature of the injector must be kept constant during the whole oven temperature program, it is preferable to use the cleaning phase instead of the transfer phase. This because during the cleaning the carrier control is managed as defined in the Carrier Gas Menu (whereas during the transfer phase the carrier control is managed as defined in the PTV control Menu).

# **Connecting PTV Backflush System**

For connecting the PTV Backflush System into the oven of the GC refer to the section **Connecting the SSL/PTV Backflush System** in the *TRACE 1300/TRACE 1310 Hardware Manual*.



# Flame Ionization Detector (FID) Module

This chapter describes the Flame Ionization Detector (FID) and provides instructions for programming the detector parameters and for performing the checkout using different injectors.

#### Contents

- Module Overview
- Operating Principle
- Gas Supplies
- Using FID Parameters
- FID Checkout

## **Module Overview**

The Flame Ionization Detector module includes the ionization cell, electrometric amplifier, heater body, and digital pneumatics for the control of the detector gases, and fittings for the connection to the analytical column.

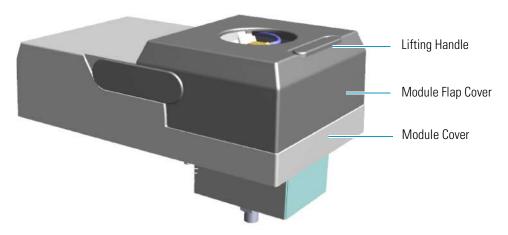


Figure 128. Flame Ionization Detector Module

The module and detector components are shown in Figure 129 and Figure 130.

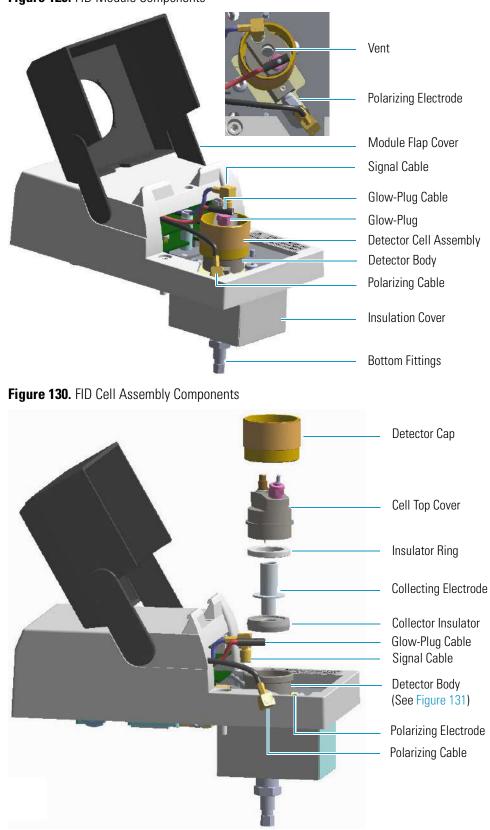


Figure 129. FID Module Components

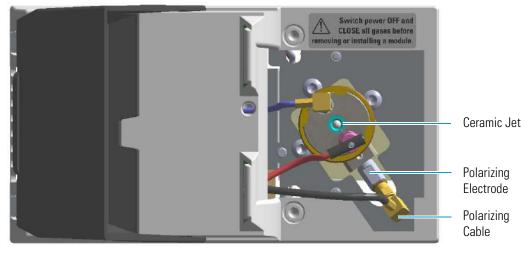


Figure 131. Ceramic Jet and Polarizing Electrode

# **Operating Principle**

The effluent from the column is mixed with hydrogen and burned in a stream of air as it emerges from the jet. The jet acts as a polarizing electrode, while a cylinder surrounding the flame forms the C. The combustion process of organic compounds creates ions that are attracted to a collector near the flame by a polarizing voltage applied between the electrodes. The resulting ionization current is sensed by an electrometer amplifier and converted to a suitable output signal.

# **Gas Supplies**

The gas flow rates must be set correctly for proper FID operation.

**CAUTION** It is your responsibility to ensure compliance with all local safety regulations concerning gas supplies.



**WARNING** Hydrogen is a potential dangerous gas. Mixed with air it may give rise to an explosive mixture. The use of hydrogen requires the operator's extreme caution due to the risks involved.

The FID uses three gases: hydrogen and air to supply the flame, and nitrogen or helium as make-up gas to enhance FID sensitivity. The stability and the analytical performance of the FID are greatly influenced by the flow of the gases through the detector.

Table 20. FID Gases (Sheet 1 of 2)

Gas	Used As:	Range	Typical Condition
Hydrogen	Fuel gas for FID flame	1-100 mL/min	35 mL/min
Air <sup>1</sup>	Fuel gas for FID flame	5-500 mL/min	350 mL/min

Gas	Used As:	Range	Typical Condition
Nitrogen	Make-up gas (recommended)	1-50 mL/min	40 mL/min
Helium	Make-up gas	1-50 mL/min	40 mL/min
<sup>1.</sup> Usually the air flow is about ten times the hydrogen flow to keep the flame lit. An excessive amount of air will			

Table 20. FID Gases (Sheet 2 of 2)

<sup>1.</sup> Usually the air flow is about ten times the hydrogen flow to keep the flame lit. An excessive amount of air wi destabilize the flame causing noise and flameout.

**Tip** To gain optimal performance from the FID, you should experiment with the hydrogen flow rate, keeping the carrier gas and air flows constant, to obtain the maximum signal intensity for the components of interest.

# **Using FID Parameters**

The following section lists and describes the parameters controlling the FID detector.

- **Temperature** Defines the temperature of the detector. Enter a temperature in the range from 0 °C to 450 °C. Enter values of 250-350 °C to prevent condensation of the water vapor formed as a result of the hydrogen combustion of the flame. Operate the detector at a higher temperature than the oven program.
- Hydrogen Flow Defines the flow of hydrogen to the detector. Acceptable values are On/Off or 1-100 mL/min. When the H<sub>2</sub> is turned on, enter the flow of Hydrogen to optimize the flame. Typical flow is 35 mL/min.
- Air Flow Defines the flow of air to the detector. Acceptable values are On/Off or 5-500 mL/min. When the air is turned On, enter the flow of Air to optimize the flame. Typical flow is 350 mL/min.
- Make-up Flow Defines the make-up flow to the detector. Nitrogen is recommended as the make-up gas to enhance FID sensitivity. Acceptable values are On/Off or 1-50 mL/min. Typical flow is 40 mL/min.
- **Flame** Ignites the flame. The range is On/Off. This can only happen if the Base Temperature is at least 150 °C and air and hydrogen flows are enabled.
- Flameout Retry When the flame ignites, the signal increases above the ignition threshold and the ignition process stops. If the signal does not increase above the ignition threshold value, the flame function attempts to ignite the FID two more times and then stops. Then the flame function indicates that the flame is off. A typical setpoint of 0.2 1.0 pA is used.
- **Ignition Threshold** Enter a value in the range from 0.0 pA to 10 pA. If the signal drops below the Ignition Threshold, it will turn on the glow-plug and attempt to light the FID. Flameout Retry routine repeats up to three times and compares the ignition threshold value to the signal pA.

• **Signal** — This parameter shows the collector current in pA (standing current level). The displayed value is also used to indicate the flame status. If the value is very low (such as 0.3 pA), the flame is off. When the value displayed is greater than the Ignition threshold, the flame is on.

### **Setting FID Parameters**

Before starting verify that the detector gases are connected, a column is correctly installed and the system is free of leaks. Check the oven and injector temperature, and the flow of the carrier gas.

- 1. Turn on the FID Temperature parameter and set the temperature to 250 °C.
- 2. Turn on and set the detector gas flows: Air at 350 mL/min, H<sub>2</sub> at 35 mL/min, Make-up at 40 mL/min.
- 3. Enable the Flame On parameter.

## **FID Checkout**

This test allows you to check instrument analytical performance after the installation has been completed. Use this procedure as a guideline, to check if your TRACE 1300/TRACE 1310 continues to perform according to the original checkout testing specifications carried out in the factory.

#### **IMPORTANT** Calculate the signal-to-noise ratio

Different chromatography data systems have generally different ways to calculate the signal-to-noise ratio. We describe a generic procedure that works for every chromatography data system.

- Choose a part of baseline without peaks or interference signals and calculate the noise over 0.1 min of acquisition. Note the noise.
- Measure the height of the peak of interest.
- Calculate the signal-to-noise ratio by dividing the peak height and the noise previously measured.
- Repeat the procedure for each peak of interest.
- To calculate the signal-to-noise ratio using a specific data system procedure, please refer to its user manual.

**Note** If it is not possible to find a suitable part of baseline to measure a suitable noise in the checkout chromatogram due to high chemical noise, acquire a baseline with the GC in stand-by at the initial temperature and measure the noise on this baseline.

#### ✤ To perform the checkout

- 1. Before starting the checkout, the following operations should be carried out:
  - a. The liner currently installed in your injector should be carefully removed and replaced with:
    - **SSL/SSLBKF/HeS-S/SL injector** 4 mm ID liner for splitless injection with the appropriate liner seal.
    - **PTV/PTVBKF injector** 2 mm ID Silcosteel liner with the appropriate liner seal.

**Note** In case of Backflush module, the Backflush metal tube must be closed with the relevant cap, having the column directly connected to the injector.

- b. A new septum should be installed properly in your injector.
- c. Verify the required gas supplies are properly connected to your instrument.
- d. The column currently installed should be carefully removed and replaced with the test column Fused Silica Capillary Column TR-5; 7 m long; 0.32 mm ID; 0.25 mm film thickness.
- e. Perform Column Evaluation and Leak Test.
- f. Verify that your data system is properly connected to your GC system.
- g. Use an automatic liquid sampler. If not, use a suitable manual 10-µL syringe.
- 2. Set the gas flows.
  - a. Use helium as carrier gas and set it to a pressure of 30 kPa in Constant Pressure Mode.
  - b. Use nitrogen as make-up gas.
  - c. Turn on and set the detector gas flows: Air at 350 mL/min, H<sub>2</sub> at 35 mL/min, Make-up at 40 mL/min.
- 3. Set the oven parameters.
  - a. Set Initial Temperature to 50 °C.
  - b. Set Initial Time to 1 minute.
  - c. Set Ramp 1 at 20 °C/min.
  - d. Set Final Temperature to 200 °C.
  - e. Set Final Time at 1 minute.
- 4. Set the injector parameters.

#### a. SSL/SSLBKF/HeS-S/SL injector

• Set Temperature at 230 °C.

- Choose operating mode **Splitless**.
- Set Splitless Time at 0.8 minutes.
- Set Split Flow at 60 mL/min.
- Turn Constant Septum Purge on.

#### b. PTV/PTVBKF injector

- Choose operating mode **PTV Splitless**.
- Set Splitless Time to 0.8 minutes.
- Set Split Flow at 60 mL/min.
- Turn Constant Septum Purge on.
- Set Inject Temperature to 50 °C.
- Set Inject Time to 0.1 minute.
- Set Transfer Ramp to 10 °C/s.
- Set Transfer Temperature to 260 °C.
- Set Transfer Time to 1 minute.
- 5. Set FID parameters.
  - a. Set Temperature to 250 °C.
  - b. Set the flame **On**.
- 6. Start the checkout
  - a. Activate your Chromatography Data System and set the parameters required for the checkout.
  - b. Perform a blank analysis injecting pure hexane.
  - c. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.
  - d. After the baseline evaluation has been completed, set-up the data system to acquire a single run.
  - e. Inject 1  $\mu$ L of test mixture (PN 33819020).

The test mixture consists of three components in n-hexane:

- Dodecane; 20 µg/mL
- Tetradecane; 20 μg/mL
- Hexadecane; 20 μg/mL
- f. Press **Start** on the GC to begin the checkout run.
- 7. The resulting chromatogram should look like the one shown in Figure 132.

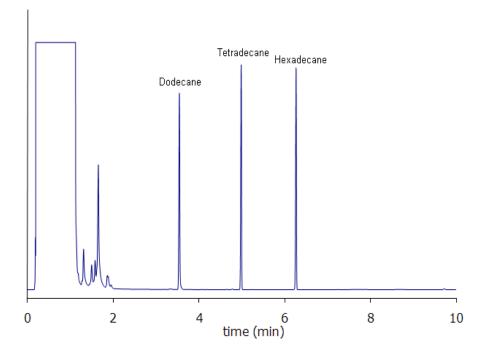


Figure 132. FID Checkout Resulting Chromatogram

The acceptance values in Table 21 indicate successful completion of the checkout. See If these criteria are not met, repeat the test.

 Table 21. FID Checkout Acceptance Values

Baseline Parameters	
Noise (fA)	<u>≤</u> 50
Drift (fA/h)	<u>≤</u> 150
Analytical Results	
Dodecane Signal-to-noise ratio	≥ 4000
Tetradecane Signal-to-noise ratio	≥ 4000
Hexadecane Signal-to-noise ratio	≥ 4000



# **Nitrogen Phosphorus Detector (NPD) Module**

This chapter describes the Nitrogen Phosphorus Detector (NPD) module, and provides instructions for programming the detector parameters and for performing the checkout using different injectors.

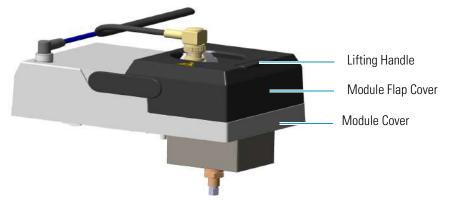
#### Contents

- Module Overview
- Operating Principle
- Source Saver
- Gas Supply
- Using NPD Parameters
- NPD Checkout

# **Module Overview**

The detector module includes the detector cell, heater body, digital pneumatics for the control of the detector gases, and fittings for the connection to the analytical column. The thermionic source is supplied by a separated power module placed into an housing on the back of the GC, or placed beside the GC, and in the most comfortable position for the user to have free access to the electrical connections.





Signal Cable Thermionic Source Assembly Cable Thermionic Source Assembly Module Flap Cover Thermionic Source Assembly Vent **Collecting Electrode Cable Bottom Fittings** Source Power Module

The module and detector components are shown in Figure 134 and Figure 135. **Figure 134.** NPD Module Components

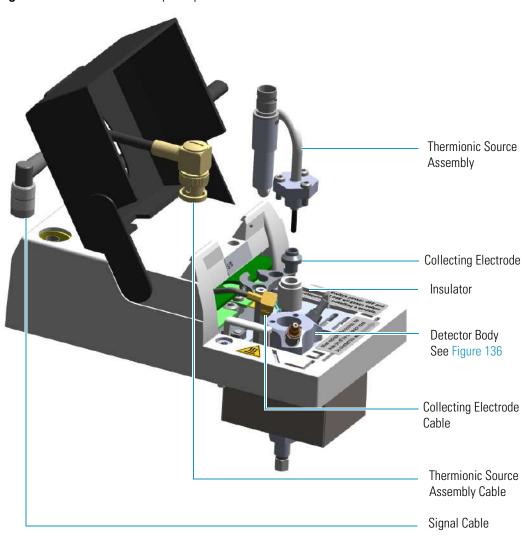
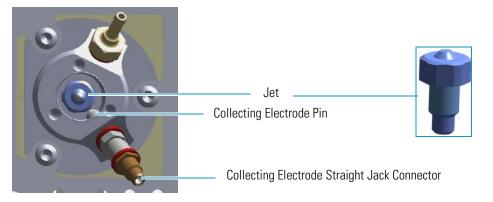


Figure 135. NPD Cell Assembly Components





# **Operating Principle**

The NPD provides selective detection of nitrogen or phosphorus-containing organic compounds. A thermionic source is electrically heated in a dilute hydrogen/air environment to create a hot chemically reactive gas layer around the source. When compounds containing nitrogen or phosphorus atoms impact this hot source, electronegative decomposition products are formed and ionized by extraction of electrons from the thermionic source.

The negative ions are then collected and detected through the electrometric amplifier. Thermionic sources with a different surface coating are available.

The lifetime of the thermionic source can vary depending on the individual source, the operating temperature, and the analytical conditions. The source heating current needs to be just high enough to produce an active layer around the source itself. When a readjustment of the source heating current is necessary, the magnitude of the detector standing current or the response to a standard sample can serve as a guide to the correct adjustment.

To prolong the source lifetime, we recommend turning off the heating current and the hydrogen flow when the detector is not being used for a prolonged period of time (for example, overnight or on weekends) or when the carrier gas flow is interrupted. Bleed from silicone-based stationary phases or residual silanizing reagents (from derivatization procedures) may contaminate the source surface with silicone dioxide and reduce the operative lifetime. Also, the extended use of halogenated solvents can adversely affect the source lifetime by the formation of reaction by-products on the source coating.

**IMPORTANT** A thermionic source type **TID 2** (black ceramic) is provided as standard with the NPD detector module for both Nitrogen and Phosphorus detections. A thermionic source type **TID 4** (white ceramic) is available as option for enhanced Nitrogen response.

For ordering an NPD thermionic source refer to the *TRACE 1300/1310 Spare Parts Guide*. For replacing a NPD thermionic source refer to the *TRACE 1300/1310 Hardware Manual*.

# **Source Saver**

The NPD module implements a functionality intended to avoid early source consumption. When the GC is idle by more than 30 minutes, to save the NPD source lifetime, the actual value of the current applied to the source bead is reduced by an amount of 10% of the source current setpoint. This will actually switch off the plasma and stops the consumption of the source itself. In the source saving condition, as soon as the GC is preparing for the next injection the source current is returned to its original setpoint. The source will switch back on and will be ready for the next analysis.

# **Gas Supply**

The gas flow rates must be set correctly for proper NPD operation.



Table 22. NPD Gases

**CAUTION** It is your responsibility to ensure compliance with all local safety regulations concerning gas supplies.

**WARNING** Hydrogen is a potential dangerous gas. Mixed with air it may give rise to an explosive mixture. The use of hydrogen requires the operator's extreme caution due to the risks involved.

The gases normally used with the NPD detector and capillary column are listed in Table 22.

Gas	Used As:	Range	Typical Condition
Hydrogen	Fuel gas for NPD plasma	0.1-10mL/min	2-4 mL/min
Air	Fuel gas for NPD plasma	5-500 mL/min	40-80 mL/min
Nitrogen	Make-up gas	1-50 mL/min	10-20 mL/min
Helium	Make-up gas	1-50 mL/min	10-20 mL/min

Nitrogen is preferred over helium because it has a much lower thermal conductivity and it requires a lower heating current for the thermionic source.

# **Using NPD Parameters**

The following section lists and describes the parameters controlling the NPD detector.

- **Source Current** Defines the current applied to heat the thermionic source. Enter a value in the range of 1.000-5.000 A.
- **Source Saver** Enables the source saver function intended to reduce the consumption of the NPD source.
- **Temperature** Defines the detector temperature. Enter a value in the range from room temperature to 450 °C.
- **Polarization Voltage** Defines the voltage applied to the collecting electrode. Enter a value in the range of 1-100 V.
- Air Flow Defines the flow of air to the detector. Acceptable values are On/Off or 5-500 mL/min. Typical flow is 50-60 mL/min.
- H2 Defines the flow of hydrogen to the detector. Acceptable values are On/Off or 0.1-10 mL/min. Typical flow is 2-3 mL/min.

• Make-up — Defines the flow of make-up to the detector. Nitrogen is recommended as the make-up gas to enhance NPD sensitivity. Acceptable values are On/Off or 1-50 mL/minute. Typical flow is 15 mL/min.

### **Setting NPD Parameters**

Before starting verify that the detector gases are connected, a column is correctly installed and the system is free of leaks. Check the oven and injector temperature, and the flow of the carrier gas.

- 1. Set the flow of the gases.
  - a. Set the Air at 60 mL/min.
  - b. Set the H2 at 2.3 mL/min.
  - c. Set the Make-up gas at 15 mL/min.
- 2. Set the detector parameter.
  - a. Set the Source Current to 2.500 then increase the current step-by step up to the ignition level.
  - b. Enable the source saver, if desired.
  - c. Set the Temperature to 300 °C.
  - d. Set the Polarization Voltage to 4 V.
- 3. Set the signal parameters.
  - a. Turn on the Digital Filter parameter if you want to reduce the response to high frequency noise and signal.
- 4. Be sure that the back-off signal is between 0 and 0.5 pA.
- 5. Switch on the source with an initial current of 2.50 A. The back-off signal can slightly increase, but should remain within 0 and 1.5 pA.
  - a. Monitor the signal through the touchscreen or through the data system, and increase the current value by steps of 0.002 A until an immediate and strong increase of the signal is observed.
  - b. Wait five minutes to let the source stabilize.
- 6. Check that source is correctly switched on.
  - a. Decrease hydrogen flow to 0.5 mL/min until the signal decreases to zero, then increase it again to its original value.
    - i. If the signal remains around zero, it means that the source is not switched on, and it is necessary to increase the current further according to the procedure just described.
    - ii. If the signal rises back to original value, it means that source is correctly switched on.

b. Increase the current value of 2% of the actual ignition current. Let the signal stabilizes until its level drops below 20 pA.



**CAUTION** Changes of gas flows and detector temperature affect the source current value required.

# **NPD Checkout**

This test allows you to check the instrument analytical performance after the installation has been completed. Use this procedure as a guideline, to check if your TRACE 1300/TRACE 1310 continues to perform according to the original checkout testing specifications carried out in the factory.

#### IMPORTANT Calculate the signal-to-noise ratio

Different chromatography data systems have generally different ways to calculate the signal-to-noise ratio. We describe a generic procedure that works for every chromatography data system.

- Choose a part of baseline without peaks or interference signals and calculate the noise over 0.1 min of acquisition. Note the noise.
- Measure the height of the peak of interest.
- Calculate the signal-to-noise ratio by dividing the peak height and the noise previously measured.
- Repeat the procedure for each peak of interest.
- To calculate the signal-to-noise ratio using a specific data system procedure, please refer to its user manual.

**Note** If it is not possible to find a suitable part of baseline to measure a suitable noise in the checkout chromatogram due to high chemical noise, acquire a baseline with the GC in stand-by at the initial temperature and measure the noise on this baseline.

#### \* To perform the NPD checkout

- 1. Before starting the checkout, the following operations should be carried out:
  - a. The liner currently installed in your injector should be carefully removed and replaced with:
    - SSL/SSLBKF/HeS-S/SL injector 4 mm ID liner for splitless injection with the appropriate liner seal.
    - **PTV/PTVBKF injector** 2 mm ID Silcosteel liner with the appropriate liner seal.

**Note** In case of Backflush module, the Backflush metal tube must be closed with the relevant cap, having the column directly connected to the injector.

- b. A new septum should be installed properly in your injector.
- c. Verify the required gas supplies are properly connected to your instrument.
- d. The column currently installed should be carefully removed and replaced with the test column Fused Silica Capillary Column TR-5; 7 m long; 0.32 mm ID; 0.25 mm film thickness.
- e. Perform Column Evaluation and Leak Test.
- f. Verify that your data system is properly connected to your GC system.
- g. Use an automatic liquid sampler. If not, use a suitable manual 10-µL syringe.
- 2. Set the gas flows.
  - a. Use helium as carrier gas and set it to a pressure of 30 kPa in Constant Pressure Mode.
  - b. Use nitrogen as make-up gas.
  - c. Turn on and set the detector gas flows: Air at 60 mL/min, H<sub>2</sub> at 2.3 mL/min, Make-up at 15 mL/min.
- 3. Set the oven parameters.
  - a. Set Initial Temperature to 70 °C.
  - b. Set Initial Time to 1 minute.
  - c. Set Ramp 1 at 20 °C/min.
  - d. Set Final Temperature to 230 °C.
  - e. Set Final Time to 1 minute.
- 4. Set injector parameters.

#### a. SSL/SSLBKF/HeS-S/SL injector

- Set Temperature to 230 °C.
- Choose operating mode **Splitless**.
- Set Splitless Time to 0.8 minutes.
- Set Split Flow at 60 mL/min.
- Turn Constant Septum Purge on.

#### b. PTV/PTVBKF injector

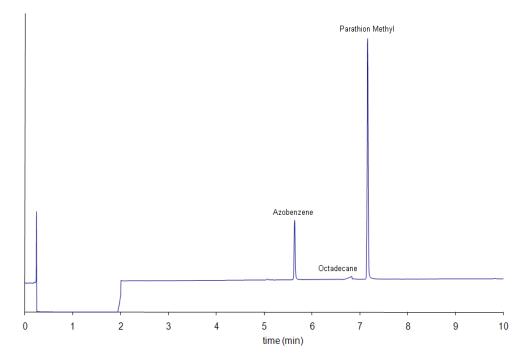
- Choose operating mode **PTV Splitless**.
- Set Splitless Time to 0.8 minutes.

- Set Split Flow at 60 mL/min.
- Turn Constant Septum Purge on.
- Set Inject Temperature to 50 °C.
- Set Inject Time to 0.1 minute.
- Set Transfer Ramp at 10 °C/s.
- Set Transfer Temperature to 260 °C.
- Set Transfer Time at 1 minute.
- 5. Set NPD parameters.
  - a. Set Temperature to 300 °C.
  - b. Set Polarizer Voltage to 4 V.
- 6. Ignite the thermionic source.
  - a. Switch on the source with an initial current of 2.50 A. The backoff signal can slightly increase, but should remain within 0 and 1.5 pA.
  - b. Monitor the signal through the keypad or through the data system, and increase the current value by steps of 0.002 A, until an immediate and strong increase of the signal is observed.
  - c. Wait five minutes to let the source stabilize.
  - d. Check that source is correctly switched on by decreasing hydrogen flow to 0.5 mL/min until the signal decreases to zero, then increase again to original value.
  - e. If the signal remains around zero, it means that the source is not switched on and it is necessary to increase further the current, accordingly to the procedure just described.
  - f. If the signal rises back to original value, it means that source is correctly switched on
  - g. Increase the current value of 2% of the actual ignition current. Let the signal stabilizes until its level drops below 20 pA.
- 7. Start the checkout.
  - a. Activate your Chromatography Data System and set the parameters required for the checkout.
  - b. Perform a blank analysis injecting pure iso-octane.
  - c. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.
  - d. After the baseline evaluation has been completed, set-up the data system to acquire a single run.
  - e. Inject 1 µL of test mixture (PN 33819006).

The test mixture consists of three components in iso-octane:

- Azobenzene; 1 μg/mL
- Octadecane; 1000 μg/mL
- Parathion Methyl; 1 μg/mL
- f. Press **Start** on the GC to begin the checkout run.
- 8. The resulting chromatogram should look like the one shown in Figure 137.

Figure 137. NPD Checkout Resulting Chromatogram



The acceptance values in Table 23 indicate successful completion of the checkout. If these criteria are not met, repeat the test.

 Table 23.
 NPD Checkout Acceptance Values

Baseline Parameters	
Noise (fA)	<u>≤</u> 50
Drift (fA/h)	≤ 200
Analytical Results	
Azobenze Signal-to-noise ratio	≥ 550
Parathion Methyl Signal-to-noise ratio	≥ 1500



# **Thermal Conductivity Detector (TCD) Module**

This chapter describes the Thermal Conductivity Detector (TCD) module and provides instructions for programming the detector parameters and for performing the checkout using different injectors.

#### Contents

- Module Overview
- Operating Principle
- Gas Selection
- Using TCD Parameters
- TCD Checkout

# **Module Overview**

The detector module includes, detector cell, heater body, digital pneumatics for the control of the detector gases, and fittings for the connection to the analytical column. The TCD cell consists of a stainless steel block containing two filaments which have the same electrical resistance, the heating elements and the temperature sensor.

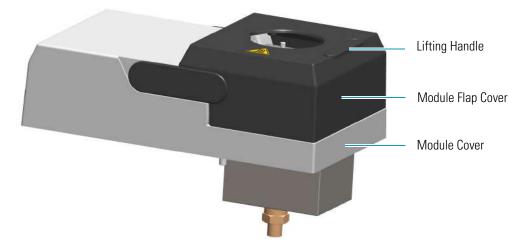
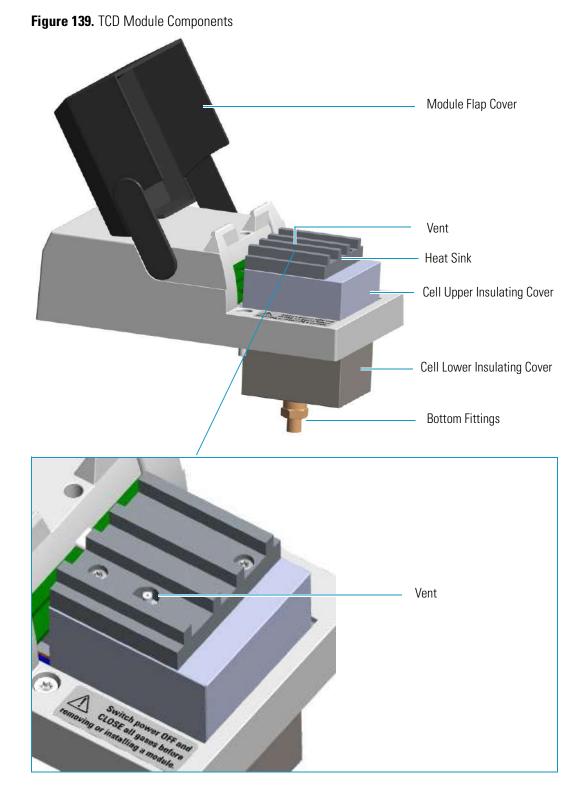


Figure 138. Thermal Conductivity Detector Module



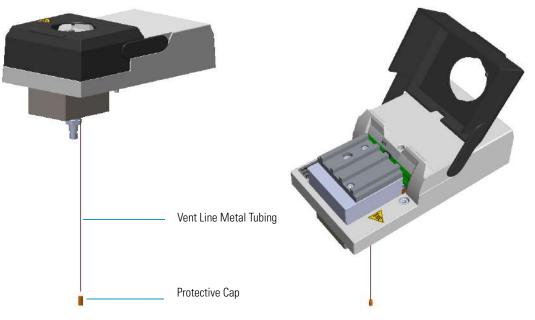
The component of module and detector are shown in Figure 139.

### **In-Series Connection TCD Module**

A modified version of the TCD module is available for the connection in-series with a second detector installed on the TRACE 1300/1310 GC, or to the methanizer in case of the TRACE 1310 Auxiliary Oven. The TCD in-series module is provided with the kit **PN 19070045**. The vent outlet of the in-series TCD module protrudes by a metal tubing (vent line) inside the GC

oven to allow further connection to a second detector or to a methanizer. See Figure 140.





**Note** For installing, connecting, and maintaining the TCD in-series module refer to the sections related to the TCD in the *TRACE 1300/1310 Hardware Manual*.

**IMPORTANT** If you are connecting the In-Series Connection TCD module to a FID, NPD, or FPD, position the vent line metal tubing so that the end of the tubing extends the proper distance above the end of the ferrule as follows:

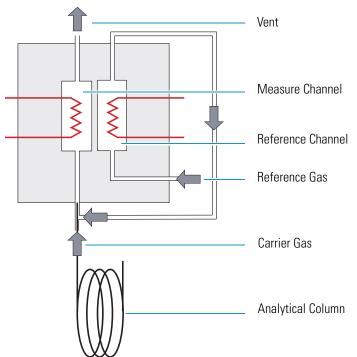
- FID, NPD = Insert the column as far as goes and withdrawn about 2 -3 mm
- FPD = 125 mm

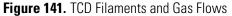
If you are connecting the In-Series Connection TCD module to an ECD, the insertion depth of vent line metal tubing must be 21 mm.

# **Operating Principle**

The TCD is sensitive to any compound having thermal conductivity other than that of the carrier gas used. The detector response depends on the relative change in the thermal conductivity of the pure carrier gas passing across the detector filament as components elute from the column. Constant filament temperature control provides a linear dynamic range. This permits the measurement of a wide range of concentrations without the need for multiple standards or sample dilution.

By measuring the amount of current required to maintain a constant temperature, as gases of varying thermal conductivities cross the filament, a chromatographic signal is produced. This process is non-destructive and is dependent on the concentration. Changes in conductivity are measured by the change in current required to keep the filament at a constant temperature.





Two flows of the same gas, carrier gas and reference gas, enter the TCD cell. The reference gas flow enters the cell, passes across the filament of the reference channel, then is mixed with the carrier gas flow coming from the injector. The resulting flow passes across the filament of the measure channel, and vents to the atmosphere.

# **Gas Selection**

The detector's response to a component is based upon the difference between the thermal conductivities of the component and the carrier gas. The greater the difference, the greater the response. The following Table 24 contains information about the thermal conductivity for a variety of gases

Gas	Thermal Conductivity ( $\lambda$ x10 <sup>7</sup> ) at 0 °C where $\lambda$ =Cal/cm x s. x °C		
Hydrogen	4130		
Helium	3363		
Methane	720		

Gas	Thermal Conductivity ( $\lambda$ x10 <sup>7</sup> ) at 0 °C where $\lambda$ =Cal/cm x s. x °C		
Oxygen	583		
Nitrogen	580		
Carbon Oxide	540		
Argon	406		
Carbon Dioxide	343		

**Table 24.** Thermal Conductivity of Some Gases (Sheet 2 of 2)

TCD requires the same gas for the measure channel (carrier gas) and the reference channel (reference gas).

- Helium is the recommended carrier gas due to its high thermal conductivity and chemical inertness.
- Low conductivity gases as argon and nitrogen, are used for special analytical requirements.
- With precautions, you can also use hydrogen as the carrier and detector gas.

**CAUTION** It is your responsibility to ensure compliance with all local safety regulations concerning gas supplies.



**WARNING** Hydrogen is a potential dangerous gas. Mixed with air it may give rise to an explosive mixture. The use of hydrogen requires the operator's extreme caution due to the risks involved.



**CAUTION** If hydrogen or helium is used, the lowest difference of temperature between the filament and cell is 50 °C. If nitrogen is used, the lowest difference of temperature between the filament and cell is 100 °C.

# **Using TCD Parameters**

The following section lists and describes the parameters controlling the TCD detector.

- **TCD Temperature** Defines the detector temperature. Enter a value in the range to 0-400 °C. A typical temperature is 200 °C depending on the application used. Set the temperature to a higher value than the maximum temperature reached by the oven during the analysis.
- Filament Power Switches on the power to the filament.
- Filament Temperature Enter a value for the constant filament temperature in the range to 50-450 °C. Filament temperatures should be kept 50-100 °C higher than the block temperature. The greater the difference the better the sensitivity. However, the usable difference between the block temperature and the filament temperature depends on the carrier gas used. The suggested  $\Delta T$  is:
  - from 50 °C to 100 °C if the carrier gas is helium.

- 100 °C if the carrier gas is nitrogen.
- Filament Max Temperature Defines the maximum limit of the filament temperature. Enter a value in the range to 50–450 °C.
- **Reference Flow** Defines the reference flow to the detector. Reference gas must be the same type of gas used for the carrier. Acceptable values are On/Off or 0.5-5.0 mL/minute. Typical flow is 1 mL/min.
- **Carrier Source** Protects the filaments when the carrier gas supply is inadvertently shut off, for example following injector septum replacement. When two injectors are present, this parameter tells the chromatograph which inlet is connected to the TCD. Select among Back, Other, Aux 1-6.
- Autozero Forces the bridge signal to zero (zeroing).
- **Negative Polarity** Inverts the polarity of the signal.

### **Selecting TCD Operating Parameters**

Program the detector temperature, the filament temperature, and the filament voltage according to the carrier gas used.

The detector sensitivity depends on the difference between the temperatures set for the detector and for the filaments: the higher the difference, the better the sensitivity. The general rule for the detector temperature is to set it higher than the maximum temperature reached by the oven during the analysis.

### **High Thermal Conductivity Gases**

When using helium or hydrogen, use the following values:

- Set the detector temperature higher than the maximum column oven temperature during the analysis.
- Set the filament temperature to 50 °C above the detector temperature.

This difference of temperature results in a high sensitivity required for trace analysis. It also ensures a longer filament lifetime.

### **Low Thermal Conductivity Gases**

When using nitrogen or argon, use the following values:

- Set the detector temperature higher than the maximum temperature reached by the column oven during the analysis, but not higher than 280–300 °C.
- Set the filament temperature to 100 °C above the detector temperature.

### **Setting TCD Parameters**

Use these general steps when using Helium as a carrier gas to analyze samples.

- 1. Set the Detector parameters.
  - a. Select TCD Temperature and enter 200 °C.
  - b. Enter 250 °C in the Filament Temperature parameter.
- 2. Set the flows parameters.
  - a. Select reference gas.
- 3. Select Negative Polarity to reverse the signal polarity.

# **TCD Checkout**

This test allows you to check the instrument analytical performance after the installation has been completed. Use this procedure as a guideline, to check if your TRACE 1300/TRACE 1310 continues to perform according to the original checkout testing specifications carried out in the factory.

#### IMPORTANT Calculate the signal-to-noise ratio

Different chromatography data systems have generally different ways to calculate the signal-to-noise ratio. We describe a generic procedure that works for every chromatography data system.

- Choose a part of baseline without peaks or interference signals and calculate the noise over 0.1 min of acquisition. Note the noise.
- Measure the height of the peak of interest.
- Calculate the signal-to-noise ratio by dividing the peak height and the noise previously measured.
- Repeat the procedure for each peak of interest.
- To calculate the signal-to-noise ratio using a specific data system procedure, please refer to its user manual.

**Note** If it is not possible to find a suitable part of baseline to measure a suitable noise in the checkout chromatogram due to high chemical noise, acquire a baseline with the GC in stand-by at the initial temperature and measure the noise on this baseline.

#### \* To perform the TCD checkout

- 1. Before starting the checkout, the following operations should be carried out:
  - a. The liner currently installed in your injector should be carefully removed and replaced with:

- SSL/SSLBKF/HeS-S/SL injector 4 mm ID liner for splitless injection with the appropriate liner seal.
- **PTV/PTVBKF injector** 2 mm ID Silcosteel liner with the appropriate liner seal.

**Note** In case of Backflush module, the Backflush metal tube must be closed with the relevant cap, having the column directly connected to the injector.

- b. A new septum should be installed properly in your injector.
- c. Verify the required gas supplies are properly connected to your instrument.
- d. The column currently installed should be carefully removed and replaced with the test column Fused Silica Capillary Column TR-5; 7 m long; 0.32 mm ID; 0.25 mm film thickness.
- e. Perform Column Evaluation and Leak Test.
- f. Verify that your data system is properly connected to your GC system.
- g. Use an automatic liquid sampler. If not, use a suitable manual 10-µL syringe.
- 2. Set the gases flow.
  - a. Use helium as carrier gas and set it to a pressure of 30 kPa in Constant Pressure Mode.
  - b. Use helium as reference gas.
  - c. Turn on and set the reference gas flow to 1 mL/min.
- 3. Set the oven parameters.
  - a. Set Initial Temperature to 50 °C.
  - b. Set Initial Time to 1 minute.
  - c. Set Ramp 1 at 20 °C/min.
  - d. Set Final Temperature to 200 °C.
  - e. Set Final Time to 1 minute.
- 4. Set injector parameters.

#### a. SSL/SSLBKF/HeS-S/SL injector

- Set Temperature to 230 °C.
- Choose operating mode **Splitless**.
- Set Splitless Time to 0.8 minutes.
- Set Split Flow at 60 mL/min.
- Turn Constant Septum Purge on.
- b. PTV/PTVBKF injector

- Choose operating mode **PTV Splitless**.
- Set Splitless Time to 0.8 minutes.
- Set Split Flow at 60 mL/min.
- Turn Constant Septum Purge on.
- Set Inject Temperature to 50 °C.
- Set Inject Time to 0.1 minute.
- Set Transfer Ramp at 10 °C/s.
- Set Transfer Temperature to 260 °C.
- Set Transfer Time to 1 minute.
- 5. Set TCD parameters.
  - a. Set TCD Temperature to 200 °C.
  - b. Set Filament Temperature to 250 °C.
  - c. Set Negative Polarity Off.
  - d. Set Filament Power On.
- 6. Start the checkout.
  - a. Activate your Chromatography Data System and set the parameters required for the checkout.
  - b. Perform a blank analysis by injecting pure hexane.
  - c. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.
  - d. After the baseline evaluation has been completed, set-up the data system to acquire a single run.
  - e. Inject 1 µL of test mixture (PN 33819016).

The test mixture consists of three components in n-hexane:

- Dodecane; 200 μg/mL
- Tetradecane; 200 μg/mL
- Hexadecane; 200 µg/mL
- f. Press **Start** on the GC to begin the checkout run.
- 7. The resulting chromatogram should look like the one shown in Figure 142.

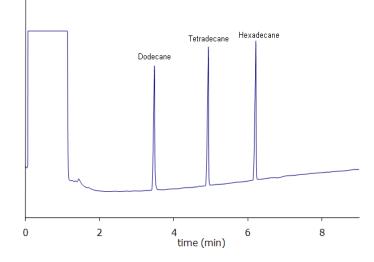


Figure 142. TCD Checkout Resulting Chromatogram

The acceptance values in Table 25 and Table 26 indicate successful completion of the checkout. If these criteria are not met, repeat the test.

Table 25. TCD with Nickel-Iron (Ni-Fe) Filament Cell Checkout Acceptance Values

Baseline Parameters	
Noise (µV)	≤ 12
Drift (µV/h)	≤ 200
Analytical Results	
Dodecane Signal-to-noise ratio	<u>≥</u> 450
Tetradecane Signal-to-noise ratio	≥ 450
Hexadecane Signal-to-noise ratio	≥ 450

Table 26. TCD with Tungsten-Rhenium (W-Re) Filament Cell Checkout Acceptance Values

Baseline Parameters	
Noise (µV)	<u>≤</u> 12
Drift (µV/h)	<u>≤</u> 200
Analytical Results	
Dodecane Signal-to-noise ratio	<u>≥</u> 100
Tetradecane Signal-to-noise ratio	≥ 100
Hexadecane Signal-to-noise ratio	<u>≥</u> 100

**Note**  $1 \mu V = 10 \mu V$  (Chrom-Card, ChromQuest)



# **Electron Capture Detector (ECD) Module**

This chapter describes the Electron Capture Detector (ECD) module and provides instructions for programming the detector parameters and for performing the checkout using different injectors.

#### Contents

- Module Overview
- Operating Principle
- Wipe Test
- Gas Supply
- Using ECD Parameters
- ECD Checkout

# **Module Overview**

The detector module includes the detector cell, heater body, digital pneumatics for the control of the detector gases, and fittings for the connection to the analytical column.

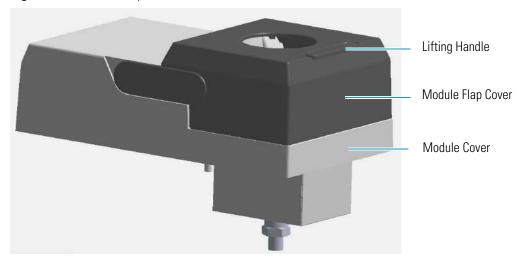
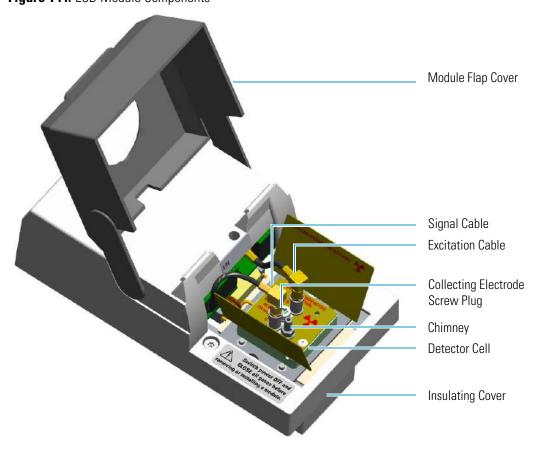
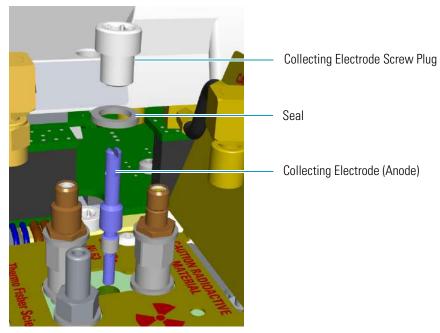


Figure 143. Electron Capture Detector Module



The components of the module and detector are shown in Figure 144 and Figure 145. **Figure 144.** ECD Module Components

Figure 145. ECD Detector Cell Components



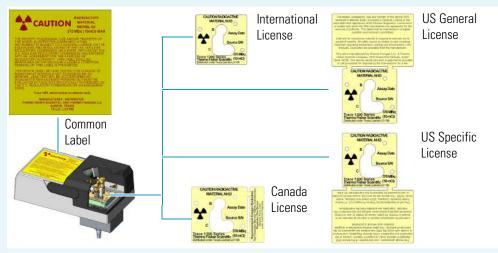
**CAUTION** The Electron Capture Detector contains a <sup>63</sup>Ni beta-emitting radioactive source at 370 MBq (10 mCi). The detector should never be opened or handled by the user. Any maintenance or service operations involving even partial disassembling of the detector must be performed ONLY by qualified personnel at a laboratory expressly authorized by Thermo Fisher Scientific and specifically licensed to handle radioactive material.

**IMPORTANT** For customers within the jurisdiction of the United States Nuclear Regulatory Commission (US NRC), you may find a listing of the agreement states and the current contact information for the regulators covering both Generally and Specifically licensed devices like an ECD at <u>http://nrc-stp.ornl.gov/rulemaking.html</u>. This information is maintained by the US NRC.

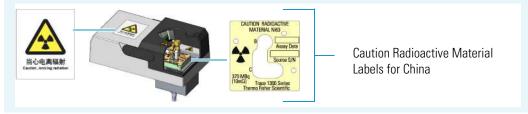
**IMPORTANT** For customers within the jurisdiction of the Canadian Nuclear Safety Commission, you may find current contact information for the regulators at <a href="http://www.nuclearsafety.gc.ca">http://www.nuclearsafety.gc.ca</a>

**IMPORTANT** The recommended working life of the detector is 15 years, after which the user should arrange for the radioactive source to be inspected and assessed by a qualified authority to extend its working life, or dispose of the product through a suitable disposal route.

**IMPORTANT** According to International, USA, and Canada licenses, the appropriate Radioactivity Data labels are applied on the ECD detector module.



Exclusively for ECD detector module used in **China**, the **Caution Radioactive Material Ni63** labels are replaced by the followings:



The maximum operating temperature of the ECD is 400 °C.



# **Operating Principle**

The detector consists of a cell containing a radioactive source, that acts as a cathode, and a collecting electrode that acts as an anode. The cell of the detector is inaccessible from the outside.

The ECD operates according to the principle of gas phase absorption of free electrons by electron capturing molecules.

The primary electrons emitted by the radioactive source (beta emission) collide with the molecules of a carrier or make-up gas (such as nitrogen) and give rise to an ionization process with the formation of secondary electrons and positive ions.

The sensitivity and selectivity of the ECD response are determined by the electron affinity of the substances entering the detection cell and are affected by the operating parameters and analytical conditions.

In the case of organic compounds, the electron affinity mainly depends on the presence of electrophores in the molecular structure as halogens, nitro groups, organo-metals, or diketons.

For halogens, the ECD response decreases in the following order: I > Br > Cl > F.

The response factor, and therefore selectivity, can vary between 1 and  $10^6$  as a function of the degree of the electron affinity of molecules. These values are also affected by temperature, which enhances the detector response for those compounds capturing electrons dissociatively.

The detector sensitivity is also affected by carrier and make-up gas flow rates, since the detector response is related to the solute concentration of the gaseous mixture.

# Wipe Test

The ECD, before leaving the factory, is tested for surface contamination by means of a **Wipe Test** (leak test) method. Each detector is provided with a certificate reporting the sequence followed and the values found.

# **Gas Supply**

The gas flow rates must be set correctly for proper ECD operation.

**CAUTION** It is your responsibility to ensure compliance with all local safety regulations concerning gas supplies.

WARNING Hydr

**WARNING** Hydrogen is a potential dangerous gas. Mixed with air it may give rise to an explosive mixture. The use of hydrogen requires the operator's extreme caution due to the risks involved.

In the ECD cell, the <sup>63</sup>Ni source releases  $\beta$  particles that collide with the molecules of an easily ionizable carrier or make-up gas flowing through the detector cell to produce low energy electrons. The commonly used gases are **nitrogen** or **argon/5% methane**. Argon/methane is recommended when a higher linear range is required or when contaminants in the carrier gas make a high mobility of electrons necessary to restore correct operating values.

Both gases should be of high purity and must not contain more than 1-2 ppm of oxygen or water vapor, since their presence would reduce the concentration of free electrons and therefore, the probability of capturing them.

The gases normally used with ECD detector are listed in Table 27.

 Table 27.
 ECD Gases

Gas	Used As:	Typical Condition
Nitrogen	Make-up gas (recommended)	15 mL/min
Argon/Methane	Make-up gas	15 mL/min

## Using ECD Parameters

The following table lists and describes the parameters controlling the ECD detector.

- **ECD Temperature** Defines the ECD detector temperature. Enter a value in the range from 0 °C to 400 °C. This temperature is generally set between 250 °C and 350 °C.
- **Reference Current** Defines the reference current of the detector. Enter a value in the range of 0.1-1.5 nA. A current value of 0.5 nA is usually applied.
- **Pulse Amplitude** Defines the voltage applied to the detector. Enter a value in the range 5 V 50 V. Typically, pulse amplitude is set to 50 V.
- **Pulse Width** Defines a value for the pulse width. The range is from 0.1  $\mu$ s to 2.0  $\mu$ s in steps of 0.1  $\mu$ s.

The value selected depends on the type of make-up gas being used with the ECD.

- Use of Nitrogen requires a pulse width value typically of  $1.0 \,\mu s$ .
- Use of Argon/Methane requires a 0.1 μs pulse to produce the highest linear range.



**WARNING** In a very clean systems the cell base frequency may fall lower than 1 kHz, being the 1 kHz frequency the optimal cell frequency. When this happens, to recover the cell frequency back to 1 kHz, it is advisable to reduce the Pulse Width by  $0.1 \,\mu s$  steps until the optimal frequency is reached.

We advise you to avoid working with ECD frequencies lower than 1 kHz.

• Make-up Flow — Specifies how much make-up gas to send to the detector. Nitrogen or Argon/Methane is suggested as the make-up gas for the ECD. The range for this entry depends on the gas being used.

The range of the make-up gas flow rates is from 1.0 mL/min to 500 mL/min for **Nitrogen** or **Argon/Methane**.

To reduce bake-out time, use higher flow rates around 60 mL/min when the detector is first turned on. Reduce the make-up gas to 15 mL/min for optimum results.

### **Setting ECD Parameters**

Before starting verify that the detector gases are connected, a column is correctly installed, and the system is free for leaks. Check oven and injector temperature, and the flow of the carrier gas.

- 1. Set the ECD Temperature to 300 °C.
- 2. Set the Reference Current to 0.5 nA.
- 3. Set the Pulse amplitude to 50 V.
- 4. Set the Pulse Width to  $1.0 \ \mu s$ .
- 5. In the Flow group box, set the make-up gas to 15 mL/min.
- 6. Allow several hours for the system to line out. The detector frequency should stabilize below 5 kHz.

## **ECD Checkout**

This test allows you to check the instrument analytical performance after the installation has been completed. Use this procedure as a guideline, to check if your TRACE 1300/TRACE 1310 continues to perform according to the original checkout testing specifications carried out in the factory.

#### **IMPORTANT** Calculate the signal-to-noise ratio

Different chromatography data systems have generally different ways to calculate the signal-to-noise ratio. We describe a generic procedure that works for every chromatography data system.

- Choose a part of baseline without peaks or interference signals and calculate the noise over 0.1 min of acquisition. Note the noise.
- Measure the height of the peak of interest.
- Calculate the signal-to-noise ratio by dividing the peak height and the noise previously measured.
- Repeat the procedure for each peak of interest.
- To calculate the signal-to-noise ratio using a specific data system procedure, please refer to its user manual.

**Note** If it is not possible to find a suitable part of baseline to measure a suitable noise in the checkout chromatogram due to high chemical noise, acquire a baseline with the GC in stand-by at the initial temperature and measure the noise on this baseline.

#### ✤ To perform the ECD checkout

- 1. Before starting the checkout, the following operations should be carried out:
  - a. The liner currently installed in your injector should be carefully removed and replaced with:
    - **SSL/SSLBKF/HeS-S/SL injector** 4 mm ID liner for splitless injection with the appropriate liner seal.
    - **PTV/PTVBKF injector** 2 mm ID Silcosteel liner with the appropriate liner seal.

**Note** In case of Backflush module, the Backflush metal tube must be closed with the relevant cap, having the column directly connected to the injector.

- b. A new septum should be installed properly in your injector.
- c. Verify the required gas supplies are properly connected to your instrument.
- d. The column currently installed should be carefully removed and replaced with the test column Fused Silica Capillary Column TR-5; 7 m long; 0.32 mm ID; 0.25 mm film thickness.
- e. Perform Column Evaluation and Leak Test.
- f. Verify that your data handling is properly connected to your GC system.
- g. Use an automatic liquid sampler. If not, use a suitable manual 10- $\mu L$  syringe.
- 2. Set the gas flows.

- a. Use helium as carrier gas and set it to a pressure of 30 kPa in Constant Pressure Mode.
- b. Use nitrogen as make-up gas and set it at 15 mL/min.
- 3. Set the oven parameters.
  - a. Set Initial Temperature to 70 °C.
  - b. Set Initial Time to 1 minute.
  - c. Set Ramp 1 at 20 °C/min.
  - d. Set Final Temperature to 220 °C.
  - e. Set Final Time to 1 minute.
- 4. Set injector parameters.

#### a. SSL/SSLBKF/HeS-S/SL injector

- Set Temperature to 230 °C.
- Choose operating mode **Splitless**.
- Set Splitless Time to 0.8 minutes.
- Set Split Flow at 60 mL/min.
- Turn Constant Septum Purge on.

#### b. PTV/PTVBKF injector

- Choose operating mode **PTV Splitless**.
- Set Splitless Time to 0.8 minutes.
- Set Split Flow at 60 mL/min.
- Turn Constant Septum Purge on.
- Set Inject Temperature to 50 °C.
- Set Inject Time to 0.1 minute.
- Set Transfer Ramp at 10 °C/s.
- Set Transfer Temperature to 260 °C.
- Set Transfer Time to 1 minute.
- 5. Set ECD parameters.
  - a. Set ECD Temperature to 300 °C.
  - b. Set Reference Current to 0.5 nA.
  - c. Set Pulse Amplitude to 50 V.
  - d. Set Pulse Width to  $1.0 \ \mu s$ .



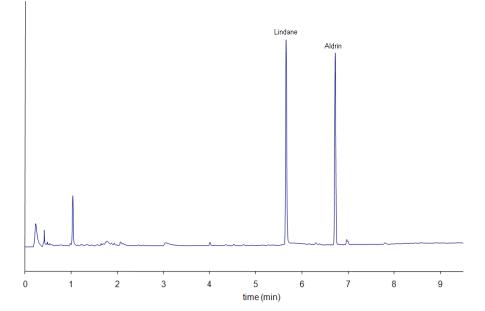
**WARNING** In a very clean systems the cell base frequency may fall lower than 1 kHz, being the 1 kHz frequency the optimal cell frequency. When this happens, to recover the cell frequency back to 1 kHz, it is advisable to reduce the Pulse Width by 0.1  $\mu$ s steps until the optimal frequency is reached. We advise you to avoid working with ECD frequencies lower than 1 kHz.

- 6. Start the checkout.
  - a. Activate your Chromatography Data System and set the parameters required for the checkout.
  - b. Perform a blank analysis injecting pure iso-octane.
  - c. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.
  - d. After the baseline evaluation has been completed, set-up the data system to acquire a single run.
  - e. Inject 1 µL of test mixture (PN 33819011).

The test mixture consists of two components in iso-octane:

- Lindane; 0.030 µg/mL
- Aldrin; 0.030 μg/mL
- f. Press **Start** on the GC to begin the checkout run.
- 7. The resulting chromatogram should look like the one shown in Figure 146.

Figure 146. ECD Checkout Resulting Chromatogram



The acceptance values in Table 28 indicate successful completion of the checkout. If these criteria are not met, repeat the test.

Table 28. ECD Checkout Acceptance Values

Baseline Parameters	
Noise (Hz)	<u>≤</u> 10
Drift (Hz/h)	<u>≤</u> 100
Analytical Results	
Lindane Signal-to-noise ratio	≥ 3000
Aldrin Signal-to-noise ratio	≥ 3000

**Note** 1 Hz = 10  $\mu$ V (Chrom-Card, ChromQuest)



# Flame Photometric Detector (FPD) Module

This chapter describes the Flame Photometric Detector (FPD) module, and provides instructions for programming the detector parameters and for performing the checkout using different injectors.

#### Contents

- Module Overview
- Operating Principle
- Gas Supply
- Using FPD Parameters
- FPD Checkout

## **Module Overview**

The detector module consists of two main components:

- 1. The FPD control module including the detector base body, the jet, the digital pneumatics for the control of the detector gases, the fittings for the connection to the analytical column, and the signal, excitation voltage and ignition/heating cables for the connection to the FPD cell assembly.
- 2. The FPD cell assembly comprises the combustion chamber, heater body, interferential filter, and photomultiplier tube for measuring the chemiluminescent emission.

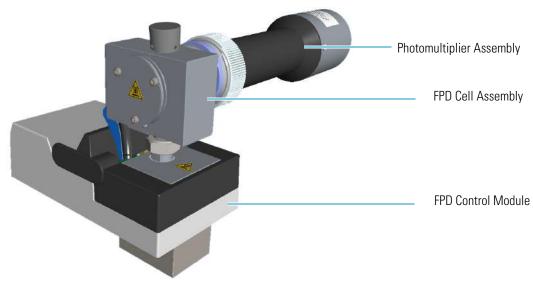
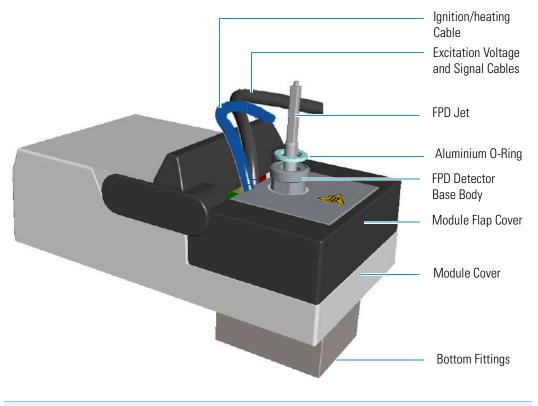


Figure 147. Flame Photometric Detector Module

The control module and detector cell assembly components are shown in Figure 148, Figure 149, and Figure 150.





**Note** The signal, excitation voltage, and ignition/heating cables will not be shown in the next illustrations for graphic convenience.

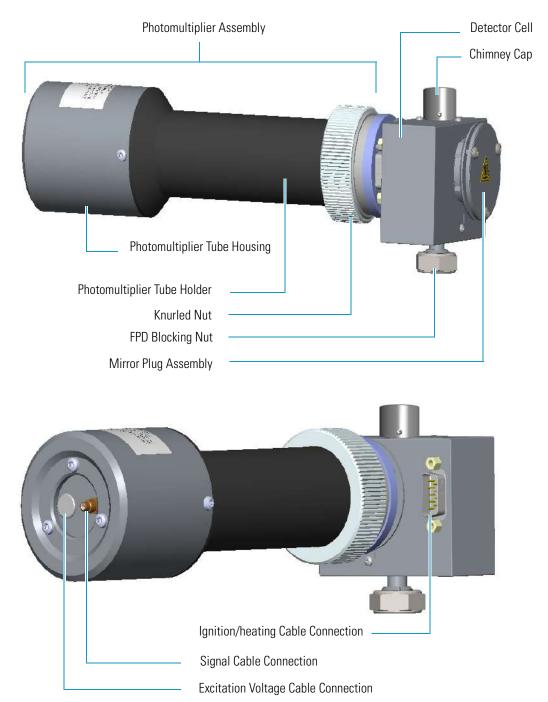
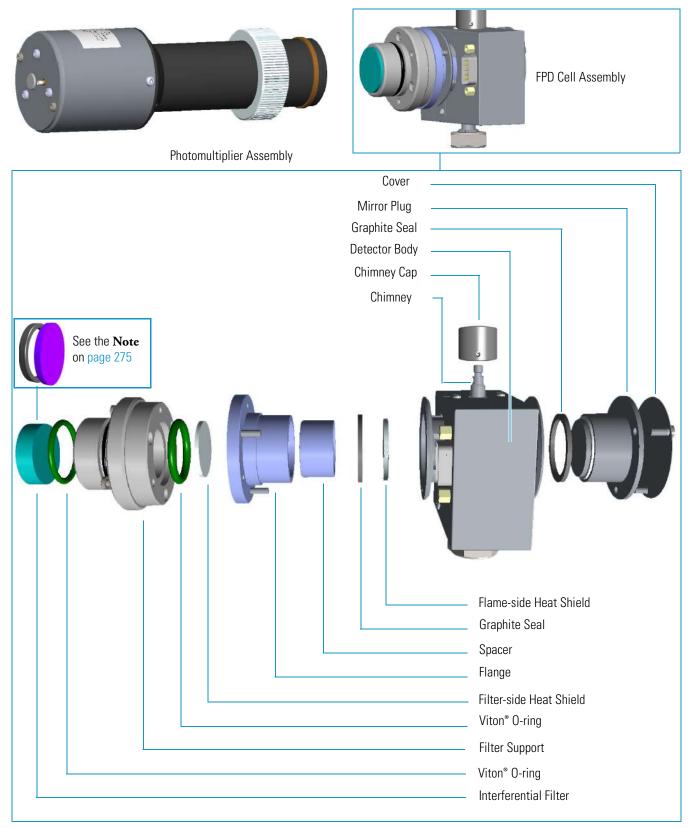


Figure 149. FPD Detector Assembly Components (Front and Back View)

#### Figure 150. FPD Cell Assembly Components



# **Operating Principle**

The FPD is based on the measurement of the characteristic radiation emitted by excited molecular species during their transition to the ground state.

Sulphur- and phosphorous-containing compounds introduced in a hydrogen rich flame decompose, giving rise to excited  $S_2^*$  and HPO\* molecular species respectively, where \* represents the excited atomic or molecular state.

The emission spectrum of  $S_2^*$  shows a maximum intensity at 394 nm while HPO\* has a maximum emission at 526 nm. These chemiluminescent emissions are isolated by appropriate narrow band optical filters and converted into measurable electrical signals by a photomultiplier tube. The interferential filter is placed between the emission chamber of the FPD and the photomultiplier tube.

There is a quadratic relationship between the number of sulphur atoms introduced in the flame and the  $S_2^*$  emission. Phosphorous compounds have a linear relationship between the HPO\* emission and the phosphorous concentration. In addition to the traditional detection of sulphur- and phosphorous-containing compounds, the FPD can be used for the selective determination of organotin compounds. In this type of application, a suitable interferential filter (610 nm) must be used. As in the phosphorous mode, the detector response is proportional to the content of heteroelement (tin) in the sample.

## Description

The FPD detector consists of a combustion chamber, a narrow band interferential filter, and a photomultiplier tube for measuring the chemiluminescent emission. The exhaust gases and the combustion products are vented through the chimney. The detector may be equipped with the sulphur filter (centered at 394 nm; PN 28107000), or the phosphorous filter (centered at 526 nm; PN 28107100). An optional tin filter (centered at 610 nm) is available with PN 28107001.

**Note** Instead of the filters PN 28107000 and PN 28107100, a new type of interferential filters for sulphur and phosphorous are available as kit:

- PN 19050785 includes an interferential filter for sulphur (PN 28107101) and a spacer.
- PN 19050786 includes an interferential filter for phosphorous (PN 28107102) and a spacer.



For replacing the interferential filter refer to the section **Maintaining a FPD** in the chapter **Performing detectors Routine Maintenance** in the *TRACE 1300/TRACE 1310 Hardware Manual.* 

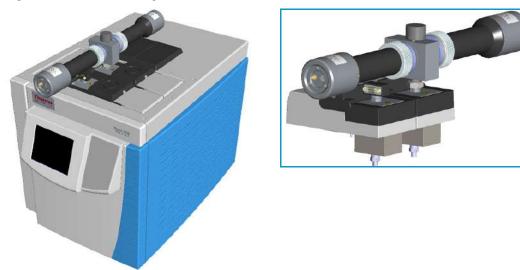
## Heating

The temperature of the cell must be sufficiently high to prevent moisture condensation. Considering that the signal to noise ratio improves by lowering the temperature of the photomultiplier tube, you should keep the FPD at relatively low temperatures (150–180 °C) and raise the base body temperature to a higher value (280–350 °C) depending on the analytical requirements.

### **Dual FPD**

The analytical capability of the Flame Photometric Detector can be expanded by connecting a second photomultiplier tube with different interferential filter on the same detector base body. See Figure 151. This configuration allows to process a sample for phosphorous and sulphur profile simultaneously, or phosphorous and tin with suitable interferential filter (610 nm). The two photomultiplier tubes, components of the FPD Dual detector, feature a single detector body in common, then the temperatures and the detector gases parameters will be the same for both.

Figure 151. Dual FPD Configuration





**IMPORTANT** To perform the Dual FPD detector configuration, you need two FPD detector modules and the kit PN 19050783. Refer to *TRACE 1300/TRACE 1310 Hardware Manual* for details.

# **Gas Supply**

The gas flow rates must be set correctly for proper FPD operation.

**CAUTION** It is your responsibility to ensure compliance with all local safety regulations concerning gas supplies.



**WARNING** Hydrogen is a potential dangerous gas. Mixed with air it may give rise to an explosive mixture. The use of hydrogen requires the operator's extreme caution due to the risks involved.

The gases normally used with the FPD detector and capillary column are listed in Table 29.

Table 29. FPD Gases

Gas	Used As:	Typical Condition
Hydrogen	Fuel gas for FPD	90 mL/min
Air	Fuel gas for FPD	115 mL/min

**Note** When operating in phosphorous mode, variations in the air/hydrogen ratio can strongly affect the response for certain phosphorous compounds, while phosphorous and sulphur containing molecules are unaffected. This characteristic allows an easy discrimination between organic phosphates and thiophosphates by simply lowering the air flow (for example, from 115 to 90 mL/min), while maintaining the same hydrogen flow rate. This possibility can be especially useful in the analysis of organophosphorous pesticide residues.

## **Using FPD Parameters**

The following section lists and describes the parameters controlling the FPD detector.

- Flame On Turns the detector ignition and detector gases on. This can only happen if the Base Temperature and the Cell Temperature are at least 150 °C, and air and hydrogen flows are enabled.
- **Base Temperature** Defines the temperature of the detector base. Enter a value in the range 0-450 °C.

**Note** To prevent stationary phase condensation from the analytical column, operate the detector at a higher temperature than the oven program. Pay special attention to the column's upper limit as specified by the manufacturer.

• **Cell Temperature** — Defines the temperature of the detector cell. Enter a value in the range 0-200 °C.



**WARNING** The default **Cell Temperature** for the FPD is **150** °**C**. This is enough to prevent water generated by the flame to condensate inside the FPD body. Higher temperatures can be used but they may have adverse effects on PMT sensitivity.

• **Ignition Threshold** — Ignition threshold is the point at which the FPD attempts to re-ignite. Set a value in the range 0.0-10.0 nA. The default value is 0.0 nA.

**Note** To select a good value for the **Ignition Threshold** parameter, the intensity of the baseline signal at flame On must be determined. Note the value of the baseline intensity at flame Off then note the value of the baseline intensity at flame On, and choose an Ignition Threshold between these two levels.

- **PMT Voltage** Defines the voltage to apply to the photomultiplier tube. If you use the default PMT voltage, the value is automatically set to 800 V, otherwise set a value in the range from 800 V to 1200 V.
- Air Defines the flow of air to the detector. Acceptable values are On/Off or 5-500 mL/min. Typical flow is 110-120 mL/min.
- **Hydrogen** Defines the flow of hydrogen to the detector. Acceptable values are On/Off or 1-100 mL/min. Typical flow is 90 mL/min.

### **Setting FPD Parameters**

Before starting verify that the detector gases are connected, a column is correctly installed and the system is free of leaks. Check the oven and injector temperature, and the flow of the carrier gas.

- 1. Set the flow of the gases.
  - a. Set the Air at 115 mL/min.
  - b. Set the H2 at 90 mL/min.
- 2. Set the detector parameters.
  - a. Set the Base temperature to 300 °C.
  - b. Set the Cell temperature to 150 °C.
  - c. Set the Ignition Threshold to 0.5 nA.
  - d. Set the PMT voltage to the default value (800 V).
  - e. Turn on the Flame On parameter.
- 3. Set the signal parameters.
  - a. Turn on the Digital Filter parameter if you want to reduce the response to high frequency noise and signal.

# **FPD Checkout**

This test allows you to check the instrument analytical performance after the installation has been completed. Use this procedure as a guideline, to check if your TRACE 1300/TRACE 1310 continues to perform according to the original checkout testing specifications carried out in the factory.

#### IMPORTANT Calculate the signal-to-noise ratio

Different chromatography data systems have generally different ways to calculate the signal-to-noise ratio. We describe a generic procedure that works for every chromatography data system.

- Choose a part of baseline without peaks or interference signals and calculate the noise over 0.1 min of acquisition. Note the noise.
- Measure the height of the peak of interest.
- Calculate the signal-to-noise ratio by dividing the peak height and the noise previously measured.
- Repeat the procedure for each peak of interest.
- To calculate the signal-to-noise ratio using a specific data system procedure, please refer to its user manual.

**Note** If it is not possible to find a suitable part of baseline to measure a suitable noise in the checkout chromatogram due to high chemical noise, acquire a baseline with the GC in stand-by at the initial temperature and measure the noise on this baseline.

#### \* To perform the FPD checkout

- 1. Before starting the checkout, the following operations should be carried out:
  - a. The liner currently installed in your injector should be carefully removed and replaced with:
    - SSL/SSLBKF/HeS-S/SL injector 4 mm ID liner for splitless injection with the appropriate liner seal.
    - **PTV/PTVBKF injector** 2 mm ID Silcosteel liner with the appropriate liner seal.

**Note** In case of Backflush module, the Backflush metal tube must be closed with the relevant cap, having the column directly connected to the injector.

- b. A new septum should be installed properly in your injector.
- c. Verify the required gas supplies are properly connected to your instrument.
- d. The column currently installed should be carefully removed and replaced with the test column Fused Silica Capillary Column TR-5; 7 m long; 0.32 mm ID; 0.25 mm film thickness.

- e. Perform Column Evaluation and Leak Test.
- f. Verify that your data system is properly connected to your GC system.
- g. Use an automatic liquid sampler. If not, use a suitable manual 10-µL syringe.
- 2. Set the gas flows:
  - a. Use helium as carrier gas and set it to a pressure of 30 kPa in Constant Pressure Mode.
  - b. Turn on and set the detector gas flows: Air at 115 mL/min, H<sub>2</sub> at 90 mL/min.
- 3. Set the oven parameters:
  - a. Set Initial Temperature to 70 °C.
  - b. Set Initial Time to 1 minute.
  - c. Set Ramp 1 at 20 °C/min.
  - d. Set Final Temperature to 230 °C.
  - e. Set Final Time to 1 minute.
- 4. Set injector parameters:
  - a. SSL/SSLBKF/HeS-S/SL injector
    - Set Temperature to 230 °C.
    - Choose operating mode **Splitless**.
    - Set Splitless Time to 0.8 minutes.
    - Set Split Flow at 60 mL/min.
    - Turn Constant Septum Purge on.

#### b. PTV/PTVBKF injector

- Choose operating mode **PTV Splitless**.
- Set Splitless Time to 0.8 minutes.
- Set Split Flow at 60 mL/min.
- Turn Constant Septum Purge on.
- Set Inject Temperature to 50 °C.
- Set Inject Time to 0.1 minute.
- Set Transfer Ramp at 10 °C/s.
- Set Transfer Temperature to 260 °C.
- Set Transfer Time at 1 minute.
- 5. Set FPD parameters:

- a. Set Base Temperature to 300 °C.
- b. Set Cell Temperature to 150 °C.
- c. Set Ignition Threshold to 0.1 nA.
- d. Set PMT voltage to default value (800 V).
- 6. Ignite the FPD flame.
  - a. Turn on the Flame On parameter. The ignition sequence starts.

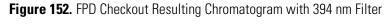
Positive variation of the signal value indicates the flame is lit. You can also verify flame ignition by holding a cold, shiny surface (such as a mirror or chrome-plated wrench) to the detector chimney vent and checking for water condensation.

After a short time, the baseline should stabilize to the standing current level of the system.

- 7. Start the checkout:
  - a. Activate your Chromatography Data System and set the parameters required for the checkout.
  - b. Perform a blank analysis injecting pure iso-octane.
  - c. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.
  - d. After the baseline evaluation has been completed, set-up the data system to acquire a single run.
  - e. Inject 1 µL of test mixture (PN 33819006).

The test mixture consists of three components in iso-octane:

- Azobenzene; 1 μg/mL
- Octadecane; 1000 μg/mL
- Parathion Methyl; 1 μg/mL
- f. Press **Start** on the GC to begin the checkout run.
- 8. The resulting chromatogram should look like the one shown in Figure 152 or Figure 153.



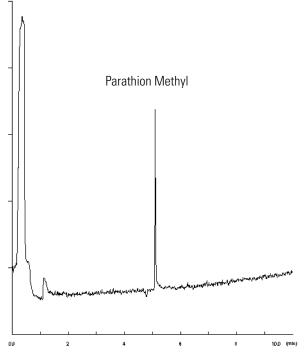
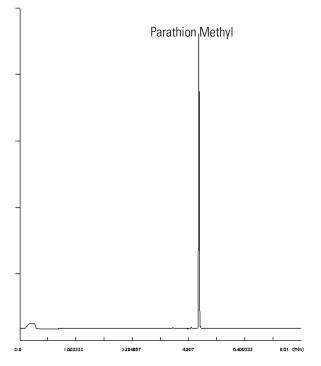


Figure 153. FPD Checkout Resulting Chromatogram with 526 nm Filter



The acceptance values in Table 30 indicate successful completion of the checkout. If these criteria are not met, repeat the test.

 Table 30.
 FPD Checkout Acceptance Values

Baseline Parameters	394 nm (S) Filter	526 nm (P) Filter
Noise (pA)	<u>&lt;</u> 600	<u>&lt;</u> 600
Drift (pA/h)	<u>&lt;</u> 1000	<u>&lt;</u> 1000
Analytical Results		
Parathion Methyl Signal-to-noise ratio	<u>≥</u> 20	<u>≥</u> 1000

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# **Instant Connect Pulsed Discharge Detector (PDD)**

This chapter describes the Instant Connect Pulsed Discharge Detector (PDD) and provides instructions for programming the detector parameters and for performing the checkout using different injectors.

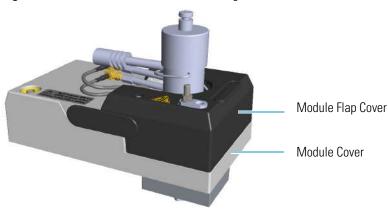
#### Contents

- Module Overview
- Operating Principle
- Gas Supplies
- Pneumatic Circuit
- Pulse Generator Voltage Rating
- Capillary and Packed Column Adapters
- Using PDD Parameters
- PDD Checkout

# **Module Overview**

The Instant Connect Pulsed Discharge Detector (PDD) module includes PDD cell, electrometric amplifier, heater body, and fittings for the connection to the analytical column. See Figure 154.

Figure 154. Instant Connect Pulsed Discharge Detector Module



A separated high voltage pulse generator supplies the pulses to the detector. The pulse generator is mounted on a support bracket hooked on the back section of the GC top cover.

**Note** The support bracket can hold two pulse generators for supplying two PDD detectors on the GC.

It is possible placing two support brackets for supplying a maximum of four PDD detectors, two installed on the GC and two installed on the TRACE 1310 Auxiliary Oven.

For further details refer to the chapter **Adding a Module** on the *TRACE 1300/1310 Hardware Manual*.

The module and pulse generator components are shown in Figure 155 and Figure 156.

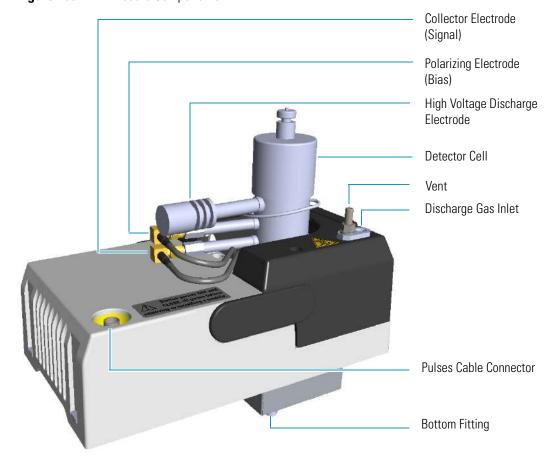
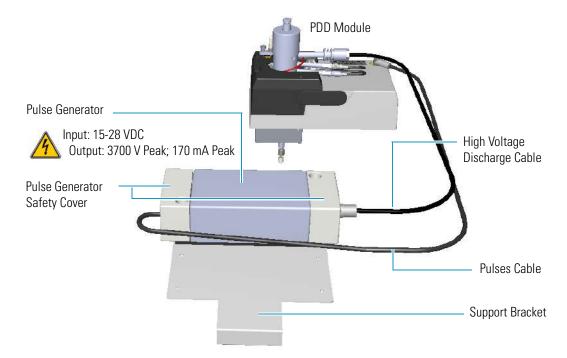


Figure 155. PDD Module Components

#### Figure 156. PDD Pulse Generator



The Instant Connect Pulsed Discharge Detector (PDD) is an universal and highly sensitive non-radioactive and non-destructive detector. It is based on the principle of the photo-ionization by radiation arising from the transition of diatomic helium to the dissociative ground state. This detector does not use radioactive sources.

The response to organic compounds is linear over five orders of magnitude with minimum detectable quantities in the low picograms range. The response to fixed gases is positive with minimum detectable quantities in the low p.p.b. range.

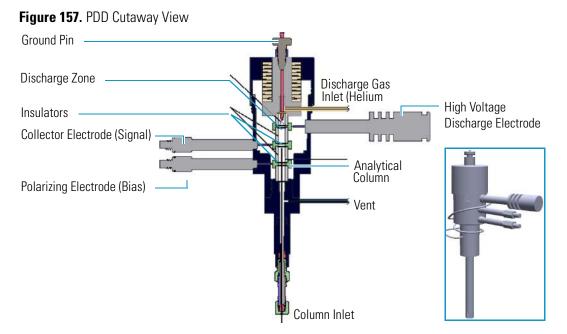
The performance of the detector is negatively affected by the presence of any impurities in the gas flows (carrier, discharge) then, the use of high quality grade of helium (99.999% pure or better) as carrier and discharge gases is strongly recommended. Because even the highest quality carrier gas may contain some water vapor and fixed gas impurities, a helium purifier is included as part of the detector system.



**IMPORTANT** If first time installation must be carry out, refer to the chapter **Adding Modules** in the *TRACE 1300/1310 Hardware Manual*.

### **Operating Principle**

PDD detector consists of a quartz cell supplied with ultra-pure helium as discharge gas that reaches the discharge zone consisting of a couple of electrodes connected to a high voltage pulses generator. See Figure 157.



The eluates from the column, flowing counter the flow of helium from the discharge zone, are ionized by photons at high energy arising from metastable Helium generated into the discharge zone. The resulting electrons are accelerated and measured as electrical signal by the collector electrode.

The discharge and carrier gas flows are opposite. For this reason it is necessary that the discharge gas flow is greater that carrier gas flow to avoid the eluates from the column to reach the discharge zone with consequent discharge electrodes contamination.

The discharge and carrier gas are flowing out together from the bottom of the cell where it is possible to measure the sum of both at the vent outlet of the detector.



**WARNING** During normal operation, the detector produce ultraviolet energy (UVA, UVB), some of which may be emitted. Do not watch the arc without eye protection.

## **Gas Supplies**



**CAUTION** It is your responsibility to ensure compliance with all local safety regulations concerning gas supplies.

The gas used for PDD discharge and carrier supply is helium.

#### **Flow Rate**

For the discharge gas an appropriate calibrated restrictor ensures a stable flow of 30 mL/min with an inlet pressure of 413 kPa (60 psig).

### **Gas Purity**

Helium must have a minimum purity of 99.999%, with < 20 ppm Ne impurity. For trace analysis of fixed gases, it is strongly recommended 99.9999% purity helium with < 0.5 ppm Ne.

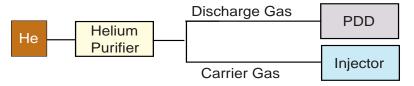


**IMPORTANT** The discharge and the carrier gases must always flow through the helium purifier.

### **Pneumatic Circuit**

Figure 158 shows the pneumatic circuit of the detector system.

Figure 158. Pneumatic Circuit



## **Pulse Generator Voltage Rating**

The voltage rating of the pulse generator is the follows:

- Input: 15-28 Vdc
- Output: 3700 V Peak; 170 mA Peak



**WARNING** Never disconnect the high voltage discharge cable when the pulse generator is turned on. Dangerous high voltage is present.

## **Capillary and Packed Column Adapters**

The PDD module is furnished with a capillary column adapter pre-installed.

**Note** For the capillary column installation length, refer to the *TRACE 1300/TRACE 1310 Hardware Manual*.

To install a packed column, the pre-installed capillary column adapter must be replaced with the **packed columns adapter** that enters into the PDD cell for the correct length.

### **Using PDD Parameters**

The following section lists and describes the parameters controlling the PDD detector.

- **Pulse Generator** Indicates the status of the pulsed discharge module. Acceptable values are On/Off.
- **PDD Temperature** Indicates the temperature of the detector. Acceptable values are from 0 to 450 °C.
- Signal (nA) Indicates the level of the standing current.

### **Setting PDD Parameters**

Before starting, verify that helium purifier and discharge gas are connected, a column is correctly installed, and the system is free of leaks. Check the oven and injector temperature, and the flow of the carrier gas.

- 1. Set the PDD Temperature according to the analytical requirement.
- 2. Turn the Pulse Generator On.
- 3. Read the Signal nA value.
  - If the system is clean, the signal value must be stabilized around 2 nA. Observe the **pink** color of the discharge generated inside the detector.
  - If a **purple** color of the discharge is observed, impurities or leaks in the discharge gas line are present.

### **PDD Checkout**

This test allows you to check instrument analytical performance after the installation has been completed. Use this procedure as a guideline, to check if your TRACE 1300/TRACE 1310 continues to perform according to the original checkout testing specifications carried out in the factory.

#### IMPORTANT Calculate the signal-to-noise ratio

Different chromatography data systems have generally different ways to calculate the signal-to-noise ratio. We describe a generic procedure that works for every chromatography data system.

- Choose a part of baseline without peaks or interference signals and calculate the noise over 0.1 min of acquisition. Note the noise.
- Measure the height of the peak of interest.
- Calculate the signal-to-noise ratio by dividing the peak height and the noise previously measured.
- Repeat the procedure for each peak of interest.
- To calculate the signal-to-noise ratio using a specific data system procedure, please refer to its user manual.

**Note** If it is not possible to find a suitable part of baseline to measure a suitable noise in the checkout chromatogram due to high chemical noise, acquire a baseline with the GC in stand-by at the initial temperature and measure the noise on this baseline.

#### \* To perform the checkout

- 1. Before starting the checkout, the following operations should be carried out:
  - a. The liner currently installed in your injector should be carefully removed and replaced with:
    - SSL/SSLBKF/HeS-S/SL injector 4 mm ID liner for splitless injection with the appropriate liner seal.
    - **PTV/PTVBKF injector** 2 mm ID Silcosteel liner with the appropriate liner seal.

**Note** In case of Backflush module, the Backflush metal tube must be closed with the relevant cap, having the column directly connected to the injector.

- b. A new septum should be installed properly in your injector.
- c. Verify the required gas supplies are properly connected to your instrument.
- d. The column currently installed should be carefully removed and replaced with the test column Fused Silica Capillary Column TR-5; 7 m long; 0.32 mm ID; 0.25 mm film thickness.

- e. Perform Column Evaluation and Leak Test.
- f. Verify that your data system is properly connected to your GC system.
- g. Use an automatic liquid sampler. If not, use a suitable manual 10-µL syringe.
- 2. Set the gas flows.
  - a. Use helium as carrier gas and set it to a pressure of 30 kPa in Constant Pressure Mode.
  - b. Use helium as discharge gas. The flow is set to 30 mL/min as fixed value.
- 3. Set the oven parameters.
  - a. Set Initial Temperature to 50 °C.
  - b. Set Initial Time to 1 minute.
  - c. Set Ramp 1 at 20 °C/min.
  - d. Set Final Temperature to 200 °C.
  - e. Set Final Time at 1 minute.
- 4. Set the injector parameters.
  - a. SSL/SSLBKF/HeS-S/SL injector
    - Set Temperature at 230 °C.
    - Choose operating mode **Splitless**.
    - Set Splitless Time at 0.8 minutes.
    - Set Split Flow at 60 mL/min.
    - Turn Constant Septum Purge on.

#### b. PTV/PTVBKF injector

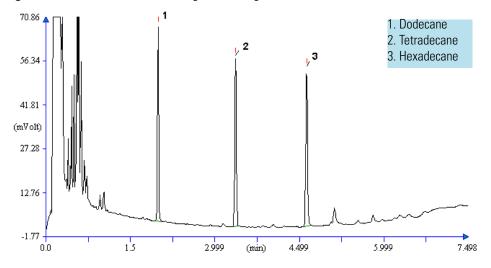
- Choose operating mode **PTV Splitless**.
- Set Splitless Time to 0.8 minutes.
- Set Split Flow at 60 mL/min.
- Turn Constant Septum Purge on.
- Set Inject Temperature to 50 °C.
- Set Inject Time to 0.1 minute.
- Set Transfer Ramp to 10 °C/s.
- Set Transfer Temperature to 260 °C.
- Set Transfer Time to 1 minute.
- 5. Set PDD parameters.

- a. Set Temperature to 300 °C.
- b. Set Pulse Generator **On**.
- 6. Start the checkout
  - a. Activate your Chromatography Data System and set the parameters required for the checkout.
  - b. Perform a blank analysis injecting pure hexane.
  - c. With the GC in Stand-by/Prep Run condition, activate the data system for 10 minutes to evaluate your baseline in isothermal condition.
  - d. After the baseline evaluation has been completed, set-up the data system to acquire a single run.
  - e. Inject 1 µL of test mixture (PN 33819032).

The test mixture consists of three components in n-hexane:

- Dodecane; 1 μg/mL
- Tetradecane; 1 μg/mL
- Hexadecane; 1 μg/mL
- f. Press **Start** on the GC to begin the checkout run.
- 7. The resulting chromatogram should look like the one shown in Figure 159.

Figure 159. PDD Checkout Resulting Chromatogram



The acceptance values in Table 31 indicate successful completion of the checkout. If these criteria are not met, repeat the test.

#### Table 31. PDD Checkout Acceptance Values

Baseline Parameters	
Noise (pA)	<u>≤</u> 10
Drift (pA/h)	<u>≤</u> 100
Analytical Results	
Dodecane Signal-to-noise ratio	<u>≥</u> 500
Tetradecane Signal-to-noise ratio	≥ 500
Hexadecane Signal-to-noise ratio	<u>≥</u> 500

**Note** 1 pA =  $10 \mu V$  (Chrom-Card, ChromQuest)



# **Generic Detector Interface (GDI)**

This chapter briefly describes the Generic Detector Interface.

#### Contents

- GDI Mechanical Module Overview
- GDI Electrical Interface Overview
- Using GDI Parameters
- Available Third-party Detector Kits

The Generic Detector Interface allows installation of third-party detectors on the TRACE 1300/TRACE 1310 GC or on the TRACE 1310 Auxiliary Oven.

To be compatible with any potential detector, the Generic Detector Interface will be customized at production/configuration time with the specific third-party detector hardware/body.

The Generic Detector Interface consists of a GDI mechanical module and a GDI electrical interface.

- The GDI mechanical module is equipped with a manifold for connecting the detector gas supplies, and with electronics for recognizing if the heater is in use or not. See "GDI Mechanical Module Overview" on page 296.
- The separated GDI electrical interface controls the pressure and the flow of up to three detector gases. The electrical interface also includes the electronics for controlling the third-party detector temperature and for the acquisition of the analog signal coming from the third-party detector. The GDI electrical interface must be placed into an housing on the back of the GC. See "" on page 298 for details.

## **GDI Mechanical Module Overview**

The GDI mechanical module components are the shown in Figure 160.

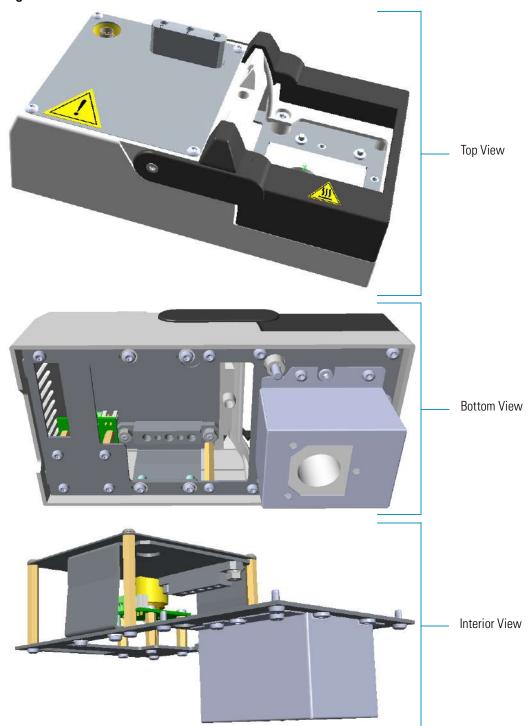


Figure 160. GDI Mechanical Module

The GDI mechanical module components are the shown in Figure 161 and Figure 162.

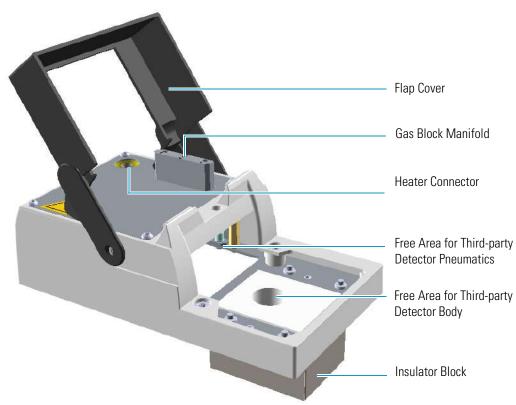
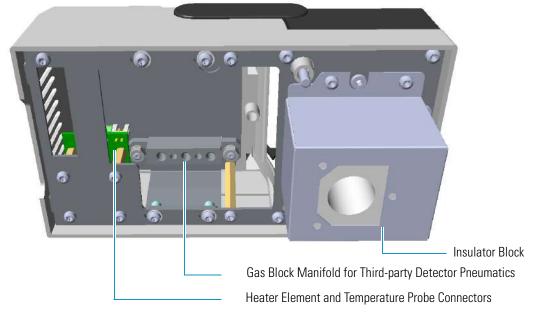


Figure 161. GDI Mechanical Module Components (1)





• If a third-party detector requires detector gases, you must carry out the specific plumbing into the free area inside the GDI mechanical module. The connections of the detector gases must be carry out to the gas block of the manifold. See the example of Figure 163.

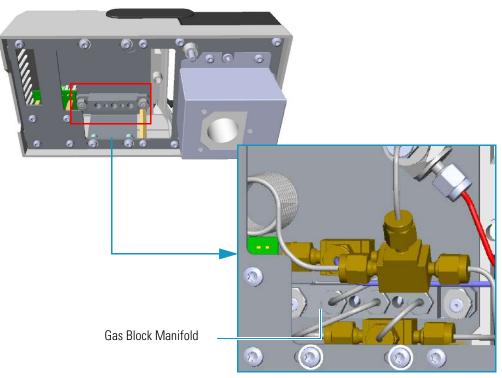
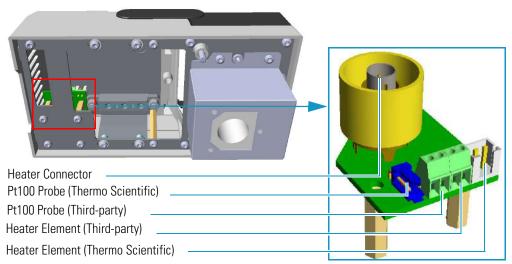


Figure 163. Example of Detector Gases Plumbing

• If a third-party detector requires the heating, you must connect the Thermo Scientific or third-party heater element and temperature probe to the heating connections inside the GDI detector module. See Figure 164.

Figure 164. Heating Connections





**WARNING** If a heater is not needed and the heated zone is not connected to an heater, the Pt100 probe must be simulated installing a 100 ohm resistor on the generic Pt100 contacts.

### **GDI Electrical Interface Overview**

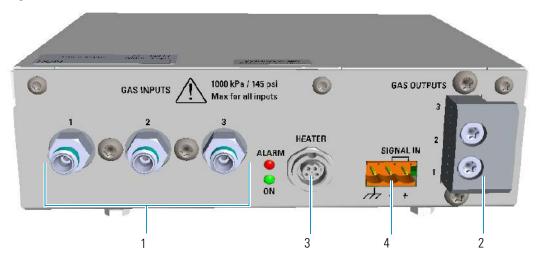
The separated GDI electrical interface controls the pressure and the flow of up to three detector gases. See Figure 165.

Figure 165. GDI Electrical Interface



The GDI electrical interface includes the following connections. See Figure 166.

Figure 166. GDI Electrical Interface Front View



1. Three inlets ports marked **Gas Inputs 1**, **2**, and **3** for the connection up to three auxiliary detector gases.



**CAUTION** The maximum pressure for all inputs is 1000 kPa (145 psi). The working inlet pressure range is from 400 kPa (58 psig) to 1000 kPa (145 psig).

2. A manifold with three outlet ports marked **Gas Outputs 1**, **2**, and **3** for the connection up to three auxiliary detector gases to the GDI mechanical module.

The GDI electrical interface factory set-up includes three flow restrictors providing a full scale of 50 mL/min of Nitrogen. All three channels **Gas1**, **Gas 2**, and **Gas 3**, are set to **Nitrogen** and 50 mL/min **full scale** by default.

If higher flow rates or very low flow rates are required, the flow restrictor must be replaced by custom one. When a custom flow restrictor is used, the new full scale must be determined.

The module is designed to use 1/4-in. encapsulated porous metal flow restrictors.

**Note** For replacing the encapsulated flow restrictor and determining the new full scale, refer to the sections **Adding a Generic Detector Interface | Replacing the Flow Restrictor** in Chapter 9 of the *TRACE 1300/TRACE 1310 Hardware Manual.* 

- 3. A 5-pin connector marked **Heater** for the connection of the heater, if required by the third-party detector, to the GDI mechanical module through the provided cable.
- 4. A 3-pin connector marked **Signal IN** for the acquisition of the analog signal coming from the third-party detector.

**Note** The detector cable with a 26-pin female connector protrudes from the back section of the GDI electrical interface. See Figure 167.

Figure 167. GDI Electrical Interface Back View



This cable will be connected to the connector **J5** (Det. Back) or **J13** (Det. Front) on the backplane board according to the Front/Back position the third-party detector is placed on the GC.

If the detector cable is not already connected into the GDI electrical interface, you must done it. Refer to the section **Adding a Generic Detector Interface** in the *TRACE* 1300/TRACE 1310 Hardware Manual for details.

Two protecting fuses are present inside the GDI electrical interface. See Figure 168 and Table 32.

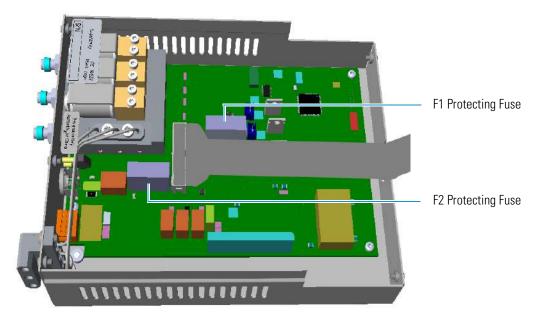


Figure 168. GDI Electrical Interface Protecting Fuses

Table 32. GDI Electrical Interface Protecting Fuses

Fuse	Туре	Protections
F1	T300 mA 250V; (5 x 20 mm)	24 Vdc
F2	T2A 250V; (5 x 20 mm)	48 Vac

### **Using GDI Parameters**

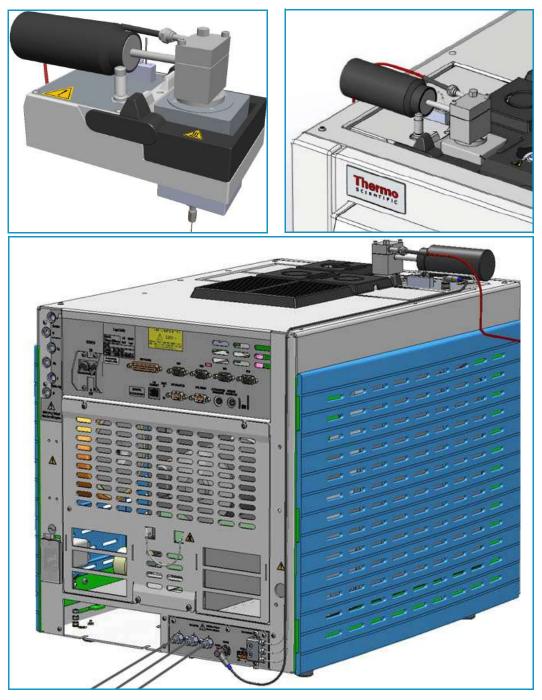
This menu includes the operating parameters for the Generic Detector Interface.

- **Temperature** Defines the third-party detector temperature in the range from room temperature to 450 °C.
- **Gas#1** Defines the **flow** of the detector gas connected to **Gas 1** line. Set the value of the gas flow in mL/min according to the **Full Scale** value of the Gas1 encapsulated flow restrictor.
- **Gas#2** Defines the **flow** of the detector gas connected to **Gas 2** line. Set the value of the gas flow in mL/min according to the **Full Scale** value of the Gas2 encapsulated flow restrictor.
- **Gas#3** Defines the **flow** of the detector gas connected to **Gas 3** line. Set the value of the gas flow in mL/min according to the **Full Scale** value of the Gas3 encapsulated flow restrictor.
- Level Shows the level of the detector signal.

# **Available Third-party Detector Kits**

The following Figure 169 shows the third-party detector Model 5380 Pulsed Flame Photometric Detector (PFPD) produced by OI Analytical<sup>™</sup> installed into a customized Generic Detector Interface, and the connections running along the GC. For specific assembling instructions, refer to the relevant User Guide.

Figure 169. Third-party PFPD Detector



# **Analog Output Interface (AOI) Module**

This chapter briefly describes the Analog Output Interface (AOI) module.

#### Contents

- Analog Output Interface Overview
- Operating Principle
- Using AOI Parameters

### **Analog Output Interface Overview**

The Analog Output Interface is an external module able to receive digital signal values from up to four detectors, and convert them to analogue signals.

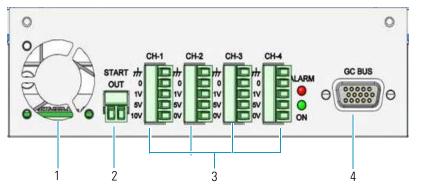
The Analog Output Interface module is placed into an housing on the back of the GC, or placed beside the GC, and in the most comfortable position for the user to have free access to the connections.

Figure 170. Analog Output Interface Module



The Analog Output Interface components are shown in Figure 171.

Figure 171. Analog Output Interface Components



The module includes the following components:

- 1. A cooling fan.
- 2. A 2-pin connector marked Start OUT.
- 3. Four channels marked CH-1, CH-2, CH-3, and CH-4, each offering: Ground (Shield), 0, 1V, 5V, and 10V.
- 4. A 15-pin female connectors marked **GC Bus** from which the Analog Output Interface takes the power and the data.

### **Operating Principle**

The analogue signal has a linear dynamic range of at least seven orders of magnitude. This is performed using four ranges changes implemented through the **timed events**.

The Analog Output Interface has a separate **Start Run** contact closure.

The Analog Output Interface is controlled only through the touch screen of the TRACE 1300/1310 GC. The Thermo Scientific chromatography data systems do not have any control over the Analog Output Interface.

The Analog Output Interface has an user interface consisting in the following elements:

- Flexible channel selection Each channel source is configurable to be one of the installed detectors. Duplicate outputs are allowed.
- Selected signal range 10<sup>0</sup> to 10<sup>3</sup> Configurable independently for each channel.
- Signal range changes Programmable from Run Time Events.
- Each channel can be **enabled/disabled** independently. A disabled channel outputs a default of 0 V.

### **Using AOI Parameters**

This menu includes the operating parameters for the Analog Output Interface module. See Figure 172.

#### Figure 172. AOI Parameters



These parameters are configured in the **Configuration** page of the TRACE 1310 user interface. See "Analog Output Interface Menu" on page 41.

Up to four channels can be selected. For each channel of interest define the position of the detector, its attenuation range, and the Out Start Run signal.

- **Det. selection** Select the position of the detector connected to the channel. Choose one: **Front** or **Back** if the detector is installed on the GC. Choose one **Left** or **Right** if the detector in installed on TRACE 1310 Auxiliary Oven.
- **Range** Select the range of the attenuation signal. Choose one: **0**, **1**, **2**, or **3**  $(10^0, 10^1, 10^2, 10^3)$ .

**Note** The attenuation signal can be modified at a given time in the **Run Table** of the GC's user interface. See Run Table on page 54.

• Out Start Run — Select how the Out Start Run signal will change. Choose High to Low or Low to High.



# **Optional Modules**

This chapter briefly describes the optional NoVent Microfluidics, FTIR Make-up, and Instant Connect Thermospray SSL Injector modules.

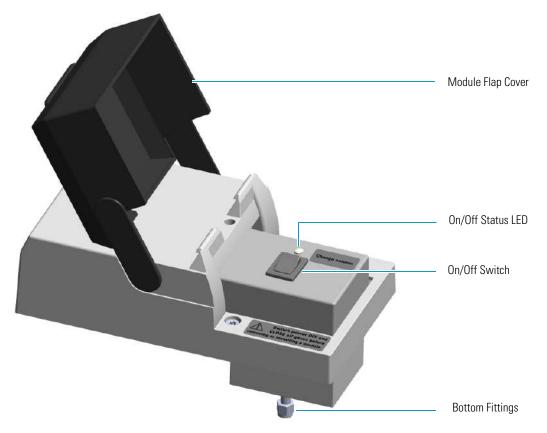
#### Contents

- NoVent Microfluidics Module
- FTIR Make-up Module
- Instant Connect Thermospray SSL Injector Module (SSI-TSI)

## **NoVent Microfluidics Module**

The NoVent Microfluidics is a solution for maintaining carrier gas flow into the mass spectrometer without breaking vacuum during column replacement, injector maintenance, or both. See Figure 173.

Figure 173. NoVent Microfluidics Module



The module includes the solenoid valve for starting and stopping the carrier gas flow, the On/Off switch, the On/Off status LED (Light Emitting Diode), and the fittings for the connection to the SilFlow<sup>TM</sup> device by using a 300 mm  $\times$  75 µm i.d. fused silica restrictor.

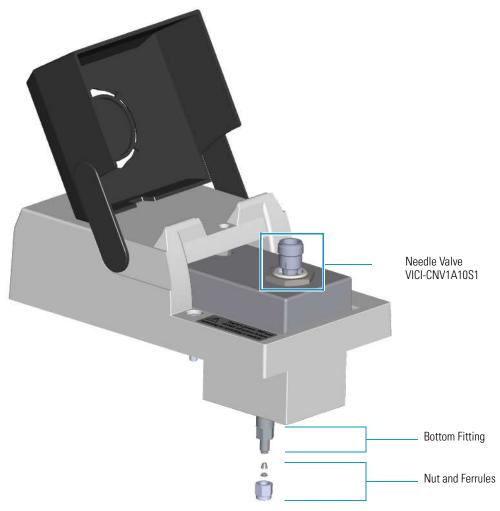
For details about the installation and the use of the NoVent Microfluidics refer to the *TRACE* 1300/TRACE 1310 Hardware Manual or to the NoVent Microfluidics Installation Guide PN 120807-0001.

### FTIR Make-up Module

The module is equipped with an On/Off needle valve for the regulation of the gas flow into the Thermo Scientific Nicolet iS50 FT-IR Spectrometer connected with the GC.

The module components are shown in Figure 174.

Figure 174. FTIR Make-up Module



The FTIR module must be installed into the detectors housing, and supplied through the line of the front/back carrier gas. If the front/back carrier gas is not already used by an injector module, you must install the front/back carrier gas inlet, and connect the carrier gas line as described in the section "**Making the Gas Supply Plumbing Connections**" on the *TRACE 1300/TRACE 1310 Hardware Manual*.

## Instant Connect Thermospray SSL Injector Module (SSI-TSI)

The Instant Connect Thermospray SSL Injector Module is designed as a "double wide" module that fits into the space provided by two injector modules (front and back) on the upper deck of a Thermo Scientific TRACE 1300/1310 series GC. See Figure 175.

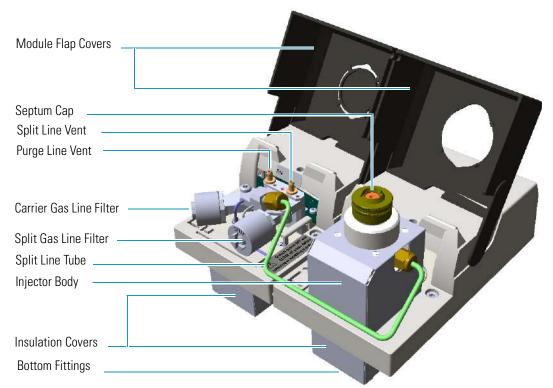


Figure 175. Thermospray SSL Injector Module

The degradation of thermolabile compounds, caused by the contact with hot surfaces, is minimized as in the thermospray technique the evaporation of the sample occurs from floating droplets. Sample is injected through **Hot Empty Needle** technique, that means needle is kept into the injector a few seconds, before pushing the syringe piston to eject the liquid sample. During this time needle becomes hot, and when the sample is ejected the evaporation starts as soon as sample pass through the hot needle.

The Thermospray SSL injector is used for split, splitless, or surged splitless injections. You can use capillary and wide-bore columns.

**Note** For the description, the programming of the injector parameters, the installation and the maintenance of the module, refer to the *Instant Connect Thermospray SSL Injector Module User Guide* PN 31709742.

# **Starting Analysis**

This chapter contains instructions for running an analysis.

#### Contents

- Confirming Your GC System is Working
- Setting Method Parameters
- Performing a Manual Injection
- Performing an Automatic Injection

With all components installed and configured, you are ready to make the first run. You will need to have your data system connected and turned on to see the chromatogram. Save the first chromatogram to compare future performance and to help resolve service issues.

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## **Confirming Your GC System is Working**

After installing a new column, you should confirm that your GC system has power, the carrier gas rate is correct, the gas tank has enough pressure, and that the system has reached temperature, and is leak-free.

### **Checking Power to the TRACE 1310**

To confirm that a TRACE 1310 is powered on, after the startup routine, make sure the front touch screen displays the Main Menu. See Figure 176.



Figure 176. TRACE 1310 Touch Screen

To power-on the GC place the power switch, located on the back side, in the On (up) position marked **I**. If the instrument still doesn't power on, check the electrical connections and wall outlet.

### **Checking Power to the TRACE 1300**

To confirm that a TRACE 1300 is powered on, make sure the Power light is on. When the GC powers on, all the LEDs of the status panel light up simultaneously, afterward the **Power** light becomes a solid green while all the other lights turn off. The GC is now in stand-by status. See Figure 177.



Figure 177. TRACE 1300 Status Panel at the GC Power On

To power-on the GC place the power switch, located on the back side, in the On (up) position marked **I**. If the instrument still does not power on, check the electrical connections and the wall outlet.

#### Verifying the Carrier Gas Flow Rate

Once you confirm that the system is powered on, you need to verify that the carrier gas rate is what you expect it to be.

#### To check the carrier gas flow rate

- 1. Access the carrier gas menu. On the TRACE 1310 choose **Instrument Control** then the **Front/Back Inlet**.
- 2. Display the column flow.
- 3. If the actual and setpoint amounts in **Column Flow** are the same, then you have good carrier gas flow. If the amounts are different, see the Analytical Troubleshooting section.

### **Checking Your Carrier Gas Tank Pressure**

Make sure you have enough pressure in the carrier gas tank to accommodate the number of samples you plan to run. If the pressure is too low, you may run out of gas in the middle of a run, which could compromise the results of your data.

#### \* To check the carrier gas tank pressure

- 1. Locate your carrier gas tank. It might be in a different room, depending on how your lab is set up.
- 2. Look at the pressure gauge on the tank.
- 3. Ensure the pressure is more than 100 psi at the primary (or first) regulator stage. If it is not, you may want to replace the tank if you have to run a lot of samples.

### **Check the Temperature**

- ✤ To check the temperature
- 1. Access the carrier gas menu. On the TRACE 1310 choose **Instrument Control** then the **Front/Back Inlet**.
- 2. Display the temperature.
- 3. If the actual and setpoint amounts in **Temperature** are the same, then you have good carrier gas flow. If the amounts are different, see the Troubleshooting section.

### **Setting Method Parameters**

An analytical method describes the treatment a sample receives during analysis. You can set parameters for:

- Temperatures, including programmed changes (ramps)
- Pressures
- Flow rates
- Inlet types
- Detector types and parameters
- Signal changes
- Autosampler parameters
- Timed events before, during, and after the run

Program the method parameters through the touch screen or through the data system.

### **Performing a Manual Injection**

#### \* To perform a manual injection

- 1. Verify that a column is correctly installed, the correct liner is in the injector, and the system is free of leaks.
- 2. Check the oven temperature and detector temperature.
- 3. Program the carrier gas flow.
- 4. Verify that you have the proper syringe for the technique you are using.
- 5. Monitor the status of the TRACE 1300/TRACE 1310 and each stage in a run through the touch screen or the status panel.

- When the **Not Ready** status is visualized, the TRACE 1300/TRACE 1310 is heating the oven to the temperature specified in the Oven menu.
- When the oven has reached the initial temperature programmed, the **Standby** status is visualized.
- 6. When the TRACE 1300/TRACE 1310 is in **Standby** status, press **Start** button to begin the **Preparation Run** during which the GC sets all conditions ready for a run.

Depending on the mode you have programmed, the TRACE 1300/TRACE 1310 will perform the following operations:

- a. When the gas saver function is programmed, **Prep Run** ends the gas saver mode and resets the split flow to the flow used during injection.
- b. In splitless mode, **Prep Run** closes the split valve and will close the septum purge valve as programmed.
- c. In surge splitless mode, **Prep Run** initiates the surge pressure.
- 7. When the **Ready to Inject** status is visualized, inject the sample:
  - a. Insert the syringe needle into the injector.
  - b. Inject the sample rapidly, and rapidly remove the syringe form the injector.
- 8. Press the **Start** button. The TRACE 1300/TRACE 1310 begins the run steps up to complete the analysis as programmed.
- 9. At the end of the run, the **Cooling** status is visualized. During it the TRACE 1300/TRACE 1310 returns to initial temperature and pressure conditions.
- 10. When the Standby status has been reached, the TRACE 1300/TRACE 1310 is ready to begins the next analytical cycle.

### **Performing an Automatic Injection**

- \* To perform an injection using an autosampler
- 1. Verify that a column is correctly installed, the correct liner is in the injector, and the system is free of leaks.
- 2. Check the oven temperature and detector temperature.
- 3. Program the carrier gas flow.
- 4. Verify that you have the proper syringe for the technique you are using.
- 5. Program the autosampler method and the samples sequence as desired.
- 6. Monitor the status of the TRACE 1300/TRACE 1310 and each stage in a run through the touch screen or the status panel.

- When the **Not Ready** status is visualized, the TRACE 1300/TRACE 1310 is heating the oven to the temperature specified in the Oven menu.
- When the oven has reached the initial temperature programmed, the **Standby** status is visualized.
- 7. When the TRACE 1300/TRACE 1310 is in **Standby** status, press the **Start** button to begin the **Preparation Run** during which the GC sets all conditions ready for a run.

Depending on the mode you have programmed, the TRACE 1300/TRACE 1310 will perform the following operations:

- a. When the gas saver function is programmed, **Prep Run** ends the gas saver mode and resets the split flow to the flow used during injection.
- b. In splitless mode, **Prep Run** closes the split valve and will close the septum purge valve as programmed.
- c. In surge splitless mode, Prep Run initiates the surge pressure.
- 8. When **Ready to Inject** status is visualized, inject the sample:
  - a. Start the sequence of sample and press **Start**. The autosampler will inject the samples according to the programmed method and sequence.
- 9. At the end of the run, the **Cooling** status is visualized where the TRACE 1300/TRACE 1310 returns to initial temperature and pressure conditions.
- 10. When the **Standby** status has been reached, the TRACE 1300/TRACE 1310 is ready to begins the next analytical cycle.



# **Analytical Troubleshooting**

This chapter describes the most common indications of a problem with a baseline, peak or result, and provides analytical troubleshooting for injectors and detectors. Use the information to troubleshoot and resolve the issue.

#### Contents

- Investigating Baseline Issues
- Investigating Peak Issues
- Investigating Results Issues
- SSL, SSLBKF, and HeS-S/SL Analytical Troubleshooting
- PTV and PTVBKF Analytical Troubleshooting
- FID Analytical Troubleshooting
- NPD Analytical Troubleshooting
- TCD Analytical Troubleshooting
- ECD Analytical Troubleshooting
- FPD Analytical Troubleshooting
- PDD Analytical Troubleshooting
- GDI Analytical Troubleshooting

For best results, use a systematic method such as the following to address problems with your results or instrument.

- Electrical Connections
  - Ensure that the electrical line is grounded and the voltage supply stable.
  - Verify that all the electrical connections between the GC and eventual subsidiary units are correct.
- Gases
  - Verify that the carrier gas line is turned on.

- Verify that the detector gases are set to the correct flow rate.
- Check that there are no leaks in the system.

#### • Injection

- Use syringes with the appropriate volume for gas chromatography.
- The sample must be prepared and diluted correctly.
- Always inject the same sample volume.
- The split ratio must remain constant.
- If present, verify the autosampler performance. When an autosampler is used, the sample vials must be correctly crimped.

#### • Chromatographic column

- The column must be appropriate for the analysis to be carried out, correctly installed and conditioned before use.
- Verify that the column-to-inlet ferrule is not leaking.
- Verify the presence of gas leaking.
- Check the column efficiency periodically.

#### • Detector

- Ensure that the detector parameters are correctly set.
- Check for eventual detector contamination.
- Data System
  - Check that the Data System in use is properly connected.

# **Investigating Baseline Issues**

 Table 33.
 Troubleshooting Baseline Issues in Your Data (Sheet 1 of 2)

Behavior	Characteristic	Cause	Remedy
Drifting	General	Stationary phase has accumulated in column.	Replace the column or cut off the end of the column.
		Carrier gas cylinder pressure too low to allow control.	Replace the carrier gas cylinder or increase the pressure.
		Drifting carrier gas or combustion gas flows.	Check the gas controllers.
		Impurities have accumulated in column.	Check impurity levels in your gas. Use the correct gas purity and filters.
	Falling	Carrier gas leak in the system.	Perform a leak test and ensure the tightness of the connections on the carrier gas line.
		Column is baking out.	Allow enough time for the column to stabilize.
	Rising	Accumulation of impurities in column.	Check impurity levels in the gas source. Use correct gas purity.
		Contaminated detector.	Check the detector and clean it.
	Rising under temperature program operation	Column contaminated.	Recondition the column.
		Carrier gas flow rate too high.	Reduce the carrier gas flow.
		Contaminated column.	Recondition or replace the column.
		Contaminated gases.	Replace gas cylinders or gas filters.
High-standing current	General	Excessive column stationary phase bleeding.	Check the oven temperature, ensuring it doesn't exceed the column upper limit. Recondition the column. Replace the column.
		Loose connections.	Ensure all interconnections and screw connections are tight.
	Dip after solvent peak	Detector contaminated.	Bake out or clean the detector.
Irregular shape	~ S-shaped	Excessive column bleed during column temperature programming.	Reduce the upper column temperature. Bake out the column. Install a high temperature column.
		Oxygen contamination is decomposing the stationary phase.	Install oxygen filters in carrier gas line.Check pneumatic and inlet systems for leaks. Use correct gas purity with low oxygen content.
	Square waves	Large AC power fluctuations; heavy equipment on same line.	Use a dedicated clean AC line of sufficient amperage

Behavior	Characteristic	Cause	Remedy
	General	Contaminated detector.	Isolate the detector from electronics. If noise disappears, clean the collecting electrode.
		Combustion-gas flow too low or too high.	Check and reset the detector-gas flows at their proper values.
		Contaminated column.	Condition the column.
		Contaminated detector gas supply.	Check the gas purity and install appropriate filters
Naina		Defective electrometer.	Replace electrometer.
Noise		Detector temperature higher than column maximum temperature.	Reduce the detector temperature to the column temperature upper limit.
	High-frequency noise	Acquisition frequency to high.	Reduce the acquisition frequency.
		External electrical interference.	Attach an AC line monitor and check purity of the AC supply
		Loose column fittings.	Tighten fittings accordingly.
		Loose detector electrical connections.	Make sure the leads are properly connected.
	General	Defective electrometer or amplifier.	Replace the electrometer or amplifier.
Spiking		Dirty detector.	Isolate the detector from the electronics. If noise disappears, clean the detector.
		External electrical interference.	Attach an AC line monitor and check the purity of the AC supply.
		Temperature too low.	Increase the temperature.

#### Table 33. Troubleshooting Baseline Issues in Your Data (Sheet 2 of 2)

# **Investigating Peak Issues**

 Table 34.
 Troubleshooting Peak Issues in Your Data (Sheet 1 of 3)

Behavior	Characteristic	Cause	Remedy
Broadening	General	Column flow too high.	Reduce the flow to slightly above optimum.
		Column flow too low.	Increase the flow to slightly above optimum.
		Split flow too low in split injection.	Increase the flow to 40-50 ml/min.
		The column performances are degraded.	Test the column at the optimum flow rate.
		Injector is dirty.	Clean or replace the liner.
		Stationary phase accumulated in the outlet.	Remove the last two coils from the column.
		Detector temperature too low.	Increase the temperature to 5°C below the column maximum.
		Injection speed too low.	Inject more rapidly in a smooth motion.
Double peaks	General	Wrong autosampler injection speed or mode.	Use a higher speed.
Fronting	General	Column or detector overloaded.	Decrease the injected amount and/or analyte concentrations. Increase the split ratio.
		Column temperature too low.	Increase the temperature.
		Stationary phase too thin.	Use a thicker-film column.
	General	Contaminated carrier gas.	Replace the cylinder or replace the filter.
		Contamination from laboratory glassware.	Ensure the glassware is clean and contamination-free.
Ghost peaks		Decomposition of injected sample.	Decrease the injection port temperature. Use the on-column injection technique.
		Dirty injection solution.	Carry out adequate clean-up of sample prior to injection.
	Broad ghost peaks	Contaminated inlet or pneumatics.	Remove the column and bake out the inlet. Use a high-quality septum. Replace the split vent filter. Install an in-line filter between the pneumatics and the inlet.
		Incomplete elution of previous sample.	Increase the final oven program temperature or total run time. Increase the column flow rate.
Irregular shape	Chair-shaped	Solvent flooding of column.	Increase the initial oven temperature.

Behavior	Characteristic	Cause	Remedy
Negative peaks	All	Integrator wires reversed.	Correct the connections.
	Some	Symptom can be normal.	
	After solvent peak	Carrier gas flow too high.	Reduce the carrier gas flow rate.
		Combustion gas flow incorrect.	Check the combustion gas flow.
		Detector contaminated.	Bake out or clean the detector.
		FID flame extinguished by solvent peak.	Check the detector temperature.
		Too much sample injected.	Inject less sample.
		The column position in SSL injector is incorrect (too high).	Check the position of the column.
No peaks		Clogged syringe needle.	Replace or repair the syringe.
	At all	Column broken or disconnected.	Check the column and connections.
		Defective electrometer or amplifier.	Replace the electrometer or amplifier.
		Defective recording device.	Replace the recording device.
		FID flame is out.	Light the flame.
		Poor or missing electrical connection.	Check the cable connections.
		The column position in SSL injector is incorrect (too high).	Check the position of the column.
	Sample peaks	Column degradation causing activity.	Inject a test mixture and evaluate the column.
		Column/oven temperature too low.	Increase the column/oven temperature. Do not exceed the recommended maximum temperature for the stationary phase.
Tailing		Dirty liner.	Clean or replace the liner.
	Sample peaks	Glass wool or inlet liner causing activity.	Replace with fresh silanized wool and a clean inlet liner.
		Inlet temperature too low.	Increase the inlet temperature.
		Poor or obstructed column connections.	Remake the column inlet connection.
		Wrong stationary phase.	Replace the column according to the column manufacturer literature.

#### Table 34. Troubleshooting Peak Issues in Your Data (Sheet 2 of 3)

Behavior	Characteristic	Cause	Remedy
		Incorrect column position in in inlet.	Reinstall the column.
Tailing <i>(Continued)</i>	Solvent peak	Initial oven temperature too high (On Column).	Reduce the initial oven temperature.
		Septum purge flow too low and/or split/splitless vent flow too low.	Check and adjust the septum purge and vent flows.
		Carrier gas flow rate too high.	Reduce the carrier gas flow rate.
		Column deteriorated.	Replace the column.
Unresolved peaks	General	Column temperature too high.	Lower the column oven temperature.
		Column too short.	Use a longer column.
		Incorrect column choice.	Install a suitable column.
		Injection technique is not adequate.	Choose a correct injection technique.

#### Table 34. Troubleshooting Peak Issues in Your Data (Sheet 3 of 3)

# **Investigating Results Issues**

 Table 35.
 Troubleshooting Results Issues in Your Data (Sheet 1 of 2)

Behavior	Characteristic	Cause	Remedy
Low reproducibility of peaks area		Concentration not compatible with the dynamic range of the detection system.	Ensure that the sample concentration is suitable for the detection system.
		Inappropriate injection technique.	Try a different injection technique.
		Injection parameters inappropriate.	Check the injection temperature and flow rates.
	General	Non reproducible sample injection technique.	Evaluate the sample preparation sequences. Compare the results with a series of standard injections.
		Leaking syringe or septum.	Check and replace the syringe and/or septum at regular intervals.
		Leaks at the injection.	Check the column connections. Run a leak check.
		Poor injection technique.	Carefully meter the injected amount. Use a clean, good-quality syringe.
		Poor split flow or ratio control.	Monitor the flow. Replace the in-line filter.
	With increased retention time	Carrier gas flow rate too low.	Increase the carrier gas flow rate. Locate and remove possible obstructions in the carrier gas line. Check the injector/column ferrules.
Poor sensitivity		Leaks in the GC carrier gas line.	Run a leak test and correct leaks.
		Syringe leaks during injection.	Replace syringe or piston seals, if applicable.
	With normal retention time	Split injection temperature too low.	Increase the temperature of the injector.

Behavior	Characteristic	Cause	Remedy
		Stationary phase deteriorated by oxygen and/or water.	Use a carrier gas free of oxygen and water.
	Decreasing	Stationary phase loss due to column bleeding.	Reduce the column temperature.
		Increasing carrier leakage.	Check the septum and column connections.
	Increasing	Carrier gas supply running out.	Replace the bottle.
Retention times		Drifting or unstable pneumatic	Monitor the column pressure or flow.
		controller.	Check and replace the controller if necessary.
	Low reproducibility	Poor injection technique.	Start the run at consistent time after injection.
		Sample size is too large.	Reduce the injected amount and/or volume.
		Unstable column temperature.	Check the front door and the cooling flap.
			Monitor the column temperature.

 Table 35.
 Troubleshooting Results Issues in Your Data (Sheet 2 of 2)

# SSL, SSLBKF, and HeS-S/SL Analytical Troubleshooting

• **Low sensitivity** is commonly caused by incorrect operating conditions. It might also be due to a dirty liner or to a liner wrongly sized for the volume of the sample injected.

Lack of sensitivity for selected compounds can also be related to discrimination phenomena induced by inadequate injection conditions.

- Possible **discrimination** of the heavy or volatile fraction of the sample may be caused by operating conditions set incorrectly with regard to the chosen injection mode. Discrimination could also be caused by:
  - degradation of the thermally labile compounds due to the catalytic effect of badly deactivated quartz wool inside the liner.
  - by-products originated in previous injections that have been not completely eliminated from the liner during the cleaning phase or the cleaning operating sequence.

A list of possible symptoms, their probable causes, and the suggested remedies when using a SSL injector are reported below.

## Discrimination of heavy compounds in splitless mode.

Possible Cause	Possible Remedy
Splitless time too short.	Set Splitless time to a higher value.
Initial temperature too high with regard to the solvent boiling point.	Set the injector temperature to a value closer to the solvent boiling point.
Flooding of the liner due to injected volume too large.	Reduce the amount of sample to be injected. Replace the liner with one of adequate diameter.
Flooding of the liner due to insufficient size.	Replace the liner with one of larger diameter.
Dirty liner.	Clean or replace the liner.
Liner not suitable for the actual sample.	Replace the liner with an adequate one. A lower diameter liner would improve the sample transfer.

## Discrimination of volatile compounds in splitless mode.

Possible Cause	Possible Remedy
Initial temperature too high.	Set the injector temperature to a lower value.
Liner diameter too small.	Replace the liner with one of larger diameter.
Volatiles are eliminated through the purge line.	Set step purge time to a value corresponding to Splitless time.

## Discrimination in split mode.

Possible Cause	Possible Remedy
Initial temperature too low.	Set the injector temperature to a higher value.
Liner diameter too large.	Replace the liner with one of smaller diameter.

## **Backflush analytical troubleshooting**

Diagnosis	Possible Cause	Possible Remedy
Tailing peaks.	Contaminated precolumn.	Verify the precolumn. Replace the precolumn.
	Leaks on the precolumn connections.	Check for leaks and rectify.
Heavy compounds are not eliminated.	Column oven temperature too high.	Reduce column oven temperature.
Presence of ghost peaks in the next run.	Column oven final temperature too low.	Increase the column oven final temperature
The sample is not completely transferred to the column.	Splitless time too low.	Increase splitless time duration.

# **PTV and PTVBKF Analytical Troubleshooting**

- Low sensitivity is commonly caused by incorrect operating conditions. It might also be due to a dirty liner or to a liner wrongly sized for the volume of the sample injected. Lack of sensitivity for selected compounds can also be related to discrimination phenomena induced by inadequate injection conditions.
- Possible **discrimination** of the heavy or volatile fraction of the sample may be caused by operating conditions set incorrectly with regard to the chosen injection mode. Discrimination could also be caused by:
  - degradation of the thermally labile compounds due to the catalytic effect of badly deactivated quartz wool inside the liner.
  - by-products originated in previous injections that have been not completely eliminated from the liner during the cleaning phase or the cleaning operating sequence.

A list of possible symptoms, their probable causes, and the suggested remedies when using a PTV injector are reported below.

Possible Cause	Possible Remedy
Splitless time too short.	Set Splitless time to a higher value.
Initial temperature too high with regard to the solvent boiling point.	Set the injector temperature to a value closer to the solvent boiling point.
Transfer temperature too low.	Set the temperature to a higher value.
Transfer time too low.	Set transfer time or cleaning time to a value close to the whole GC run time.
Flooding of the liner due to injected volume too large.	Reduce the amount of sample to be injected. Replace the liner with one of adequate diameter.
Flooding of the liner due to insufficient size.	Replace the liner with one of larger diameter.
Dirty liner.	Clean or replace the liner.
Liner not suitable for the actual sample.	Replace the liner with an adequate one. A lower diameter liner would improve the sample transfer.
Quartz wool causing excessive retention of high molecular compounds.	Replace the packed liner with a new one filled will quartz wool. Replace the packed liner with an empty one.

#### Discrimination of heavy compounds in splitless mode.

## Discrimination of volatile compounds.

Possible Cause	Possible Remedy
Injection speed set too low.	Set Inject speed to a higher value.
Initial temperature set too high.	Set Inject temperature to a lower value.
Split flow set too high.	Lower the split flow.
Lack of an adequate quartz wool packing inside the liner.	Fix the quartz wool packing inside the liner. Replace the liner with another filled with quartz wool.

## Discrimination of volatile compounds in splitless mode.

Possible Cause	Possible Remedy
Initial temperature too high.	Set the injector temperature to a lower value.
Liner diameter too small.	Replace the liner with one of larger diameter.
Volatiles are eliminated through the purge line.	Set step purge time to a value corresponding to Splitless time.

## Discrimination of volatile compounds in large volume mode.

Possible Cause	Possible Remedy
Initial temperature too high.	Set injector temperature to a lower value.
Injection and evaporation time are set as to exceed the time required for the solvent elimination.	Set Inject time and Evaporation time to adequate values.
Split flow set too high.	Set Split flow to a lower value.
Lack of an adequate quartz wool packing inside the liner.	Insert quartz wool in the liner.

## Discrimination split mode.

Possible Cause	Possible Remedy
Transfer temperature too low.	Set temperature to a higher value.
Transfer time too low.	Set transfer time or cleaning time to a value close to the whole GC run time.
Initial temperature too low.	Set injector temperature to an higher value.
Injected volume too large.	Reduce the amount of sample to be injected. Replace the liner with the 2 mm ID version.

## Excessive broadening of the solvent peak.

Possible Cause	Possible Remedy
Injection pressure set too high.	Set Inject pressure to a lower value.

## Volatile compound obscured.

Possible Cause	Possible Remedy
Injection pressure set too high.	Set Inject pressure to a lower value.

## Lack of sensitivity.

Possible Cause	Possible Remedy
Injection pressure set too high.	Set Inject pressure to a lower value.

## Sample degradation.

Possible Cause	Possible Remedy
Dirty liner.	Clean or replace the liner.
Transfer temperature set too high.	Set temperature to a value adequate to the nature of the sample compounds.
Liner diameter too large.	Replace the liner with one of smaller diameter to improve transfer efficiency.
Cathalytic and thermal degradation of sensitive compounds caused by the quartz wool inside the liner.	Use a new packed liner. If the symptom does not disappear, use an empty liner.

## Poor reproducibility of results.

Possible Cause	Possible Remedy
Transfer temperature too low.	Set temperature to a higher value.
Transfer time too low.	Set transfer time or cleaning time to a value close to the whole GC run time.
Injection speed set too high.	Reduce injection speed.
Injected volume too large.	Reduce the amount of sample to be injected.
Initial temperature set too low.	Set inject temperature to an higher value.
Injection pressure set too high.	Set Inject pressure to a lower value.
Lack of an adequate quartz wool packing inside the liner.	Fix the quartz wool packing inside the liner. Replace the liner with another filled with quartz wool.

## **Backflush analytical troubleshooting**

Diagnosis	Possible Cause	Possible Remedy
Tailing peaks.	Contaminated precolumn.	Verify the precolumn. Replace the precolumn.
	Leaks on the precolumn connections.	Check for leaks and rectify.
	Backflush starts too late due to Transfer time too long.	Reduce transfer time duration.
Heavy compounds are not eliminated.	Column oven temperature too high.	Reduce column oven temperature.
	Cleaning flow or cleaning time too low.	Increase cleaning flow or cleaning time or cleaning temperature.
Presence of ghost peaks in the next run.	Column oven final temperature too low.	Increase the column oven final temperature
Too solvent into the column during PTV	Backflush not enabled Incorrect transfer time or evaporation time duration.	Enable Backflush. Set sufficient time duration.
Large Volume run.	Vent flow too low respect to the used solvent.	Optimize injector temperature and vent flow.
The sample is not completely transferred to the column.	Splitless time too low.	Increase splitless time duration.

# **FID Analytical Troubleshooting**

• **Change in sensitivity**—The sensitivity of the Flame Ionization Detector depends on the carrier and detector gases flow rates and to the detector temperature.

If increased FID noise or loss in sensitivity is found, ensure the correct purity of the gases used and check traps and filters in the gas supply lines. Clean the collecting electrode and/or the jet that could be dirty. Replace them if necessary.

• **Gases**—The carrier gas flow range depends on the type of the gas used and on the type and diameter of the capillary column installed.

The hydrogen flow rate has been experimentally optimized to gain optimum analytical performance from the FID. Incorrect hydrogen and make-up flow rates reduce the FID sensitivity. The flow rate of the air is less critical than the hydrogen one. An excessive amount of air will destabilize the flame, causing noise and eventual flameout. A low flow rate of air will reduce the detector sensitivity.

**Note** Generally the air flow rate must be set to about 10 times the hydrogen flow rate to keep the flame lit.

- **Contamination**—For high sensitivity applications, it is essential that you exclude all traces of organic contamination from the chromatographic system and/or from the gas lines of the detector. Such contamination would cause ghost peaks in the chromatogram or, more commonly, an unstable baseline.
- **Condensation**—It is the result of the water vapor formed by the combustion process of the flame. The detector temperature should be normally set to the upper temperature limit of the column in use. To avoid contamination and possible rusting do not set a temperature of less than 150 °C.
- Flame Ignition—You can ignite the flame as soon as the detector temperature has reached 150 °C. After the flame appears to have ignited, a signal > 1.5 pA should be read.

**Tip** If you have a doubt that the flame is ignited, check for water vapor condensed on a mirror or on the polished end of a wrench directly over the FID vent. You should observe steam condensing on the cold surface. If not, the flame is not ignited.



**CAUTION** Do not lean over the FID to see the flame, it is invisible.

The possible causes of flame ignition problems and the relevant remedies are reported below.

Possible Cause	Possible Remedy
Incorrect fuel gas flows.	Make sure that all flows are correct measuring the hydrogen, air and make-up flow rates.
Defective igniter.	Check the glow-plug element.
Broken or cracked flame jet.	Replace the jet.
Blocked jet tip.	Check for a blocked jet by measuring the hydrogen flow with a flowmeter. If required, remove and clean the jet.
Faulty electronics.	Contact your local Thermo Fisher Scientific customer support organization.
Contamination.	Clean the detector. Contact your local Thermo Fisher Scientific customer support organization.

#### Flame ignition issues.

# **NPD Analytical Troubleshooting**

The main cause of changes in **sensitivity** is related to the thermionic source. Its gradual depletion results in a drop of sensitivity, can be compensated for by increasing the source current. Take into account that increasing the current results in a shorter source life. Loss of sensitivity is also related to source contamination, due to high boiling sample compounds that are not completely eliminated. Changes in detector temperature can also reduce sensitivity. A list of possible symptoms, their probable causes, and the suggested remedies when using an NPD detector are reported below.

#### No NPD response.

Possible Cause	Possible Remedy
Source heating current too low.	Increase the heating current.
No hydrogen flow.	Turn the hydrogen flow on and set it to a correct value.
No air flow.	Turn the air flow on and set it to a correct value.
Source turned off.	Turn the source on.
Source faulty.	Replace the source.

#### NPD response lower than expected.

Possible Cause	Possible Remedy
Lower source temperature.	Check the temperature and source heating current.
Air contamination in hydrogen line.	Turn the source off. Increase the hydrogen pressure for 10-20 minutes to purge the hydrogen line. Check the line tightness.

#### High background level.

Possible Cause	Possible Remedy
Heating current too high.	Set the correct operating parameter.
Hydrogen flow too high.	Set the correct operating parameter.
Air and/or Make-up gas flow too low.	Set the correct operating parameter.
Excessive column bleed.	Column conditioning has to be performed.

#### NPD-like response for solvent and other carbon based compounds.

Possible Cause	Possible Remedy
Hydrogen flow too high.	Reduce hydrogen flow to a lower value.

## Solvent quenching effect.

Large negative baseline upset at solvent elution with no recovery to the original baseline.

Possible Cause	Possible Remedy
Heating current too low.	Slightly increase the source heating current.

#### Unstable baseline.

Possible Cause	Possible Remedy
Background current level too high.	Reduce the source heating current value.
Hydrogen flow fluctuation.	Check pressure regulators on hydrogen line.

#### Low carbon rejection.

Possible Cause	Possible Remedy
Hydrogen flow too high.	Reduce hydrogen flow to the correct operative conditions.

# **TCD Analytical Troubleshooting**

- Sensitivity is related to the detector temperature (increasing temperature reduce sensitivity) and to the flow rate of the carrier, reference, and make-up gas. A gain in sensitivity can be obtained by increasing the gap between the temperature of the TCD body and that of the filaments. Sensitivity is strictly related to the state of the filaments and to their operating conditions. A significant reduction of the detector sensitivity may be caused by contamination of the filaments, due to degradation of high molecular weight compounds inside the cell, or to contaminated gases. Low temperatures of the detector block may cause high boiling compounds to condense on the filaments reducing sensitivity.
- A small **baseline drift** normally occurs during a temperature program and does not indicate a problem. This effect is due to the decrease of the carrier gas flow rate as temperature increases. A baseline that suddenly goes out of scale and a rapid growth of the signal could indicate that filaments are likely to be broken and must be replaced.
- **Negative peaks** are normally generated by sample components that have a thermal conductivity higher than the carrier gas. For instance, when using nitrogen or argon as carrier gas, negative peaks are obtained for helium, hydrogen or methane.

A list of possible symptoms, their probable causes, and the suggested remedies when using an TCD detector are reported below.

## TCD does not work.

Possible Cause	Possible Remedy
Filament power Off.	Lack of carrier, make-up gas or pressure too low. Check the feed of the carrier, make-up and reference gas.
Filament broken.	Contact your local Thermo Fisher Scientific customer support organization.

# **Baseline fluctuation.**

Possible Cause	Possible Remedy
Unstable regulation of the flow rate of the gases.	Check the controllers of the carrier, and make-up gases work well.
Leaks on the gas lines.	Check that the connections of the carrier, and make-up gas are tight.
Inlet pressure of gases is too low.	Set the pressure of the carrier, and make-up gas to a proper value.
Faulty temperature regulation.	Contact your local Thermo Fisher Scientific customer support organization.

## Baseline drift.

Possible Cause	Possible Remedy
Unstable regulation of the flow rate of the gases.	Check the controllers of the carrier, reference, and make-up gases work well.
Leaks on the gas lines.	Check the connections of the carrier, reference and make-up gas are tight.
Column conditioning not correctly performed.	Recondition the column according to the manufacturer's instructions.
Injector septum bleeding.	Check the operating temperature of the septum is adequate. Replace the septum if necessary.
Faulty temperature control.	Contact your local Thermo Fisher Scientific customer support organization.

# Low Sensitivity.

Possible Cause	Possible Remedy
Leaks on the gas lines.	Check the connections of the carrier, reference and make-up gas are tight.
Leak due to septum wearing.	Replace the septum.
Operating conditions of the detector not properly set.	Optimize working parameters of the detector according to the actual operating mode (Constant Voltage, Constant Temperature).

Possible Cause	Possible Remedy
Thermal conductivity of the carrier gas is too close to the one of the compounds to be analyzed.	Use a different carrier gas.
Contaminated filaments.	Remove contamination by baking the filaments for one hour at a temperature higher than the boiling point of the most high-boiling compound. Perform baking twice if necessary. If the symptom does not disappears, contact your customer support organization.
Oxidized filaments.	Contact your local Thermo Fisher Scientific customer support organization.

# **ECD Analytical Troubleshooting**

A list of possible symptoms, their probable causes, and the suggested remedies when using an ECD detector are reported below.

## High baseline frequency.

Possible Cause	Possible Remedy
Impure gas supply.	Use high purity grade gases and filters to trap moisture and oxygen.
No or insufficient flow of make-up gas.	Increase make-up gas flow rate.
Excess column bleed.	Column conditioning is needed.
Leaks on carrier and/or make-up gas lines.	Run a leak test.
Chemical contaminated collecting electrode.	Clean the collecting electrode.
Chemically contaminated radioactive source.	Contact your customer support organization.
Pulse width not correctly set.	Set Pulse width to 1.0 $\mu s$ for Nitrogen and 0.1 $\mu s$ for Ar/CH_4.

## Negative dips after peaks.

Possible Cause	Possible Remedy
Chemically contaminated collecting electrode.	Clean the collecting electrode.
Chemically contaminated radioactive source.	Contact your local Thermo Fisher Scientific customer support organization.

## Baseline drift with changing pulse voltage.

Possible Cause	Possible Remedy
Chemically contaminated collecting electrode.	Clean the collecting electrode.

## High noise

Possible Cause	Possible Remedy
The system is clean, the frequency is low but the noise is high.	In a very clean systems the cell base frequency may fall lower than 1 kHz, being the 1 kHz frequency the optimal cell frequency. When this happens, to recover the cell frequency back to 1 kHz, it is advisable to reduce the Pulse Width by 0.1 µs steps until the optimal frequency is reached. We advise to avoid working with ECD frequencies lower than 1 kHz.

# **FPD Analytical Troubleshooting**

• **Sensitivity** The FPD sensitivity is affected by the temperature. The FPD response decreases with an increase in the detector temperature. In addition, the detector response can be severely reduced if a non-sulphur or non-phosphorus compound is partially or fully eluted with the analyte of interest (**quenching**).

Keep clean the interferential filter and the heat shields as described in the maintenance sequences, to avoid reducing sensitivity. Replace the heat shields if the deposits due to contamination cannot be eliminated.

• **Gases** A right choice of the flow rates of hydrogen and air as fuel gases is of primary importance in determining FPD sensitivity. Flow rates will affect also selectivity and, to a great extent, peak shapes.

A list of possible symptoms, their probable causes, and the suggested remedies when using an FPD detector are reported below.

#### High standing current and noise.

Possible Cause	Possible Remedy
Detector conditioning not completed	Increase detector conditioning time
Air flow rate set too high	Reduce air flow rate
Detector not light-tight	Tighten the chimney cap and the knurled nut that fixes the photomultiplier tube
Interferential filter missing	Check the interferential filter has been correctly installed

#### No standing current.

Possible Cause	Possible Remedy
Signal or power cables incorrectly connected	Check connections
Faulty photomultiplier tube	Contact your local Thermo Fisher Scientific customer support organization.

## Unstable and excessively noisy baseline.

Possible Cause	Possible Remedy
Faulty photomultiplier tube	Contact your local Thermo Fisher Scientific customer support organization.

#### FPD temperature does not reach the setpoint.

Possible Cause	Possible Remedy
Power cable not properly connected	Check cable connection
Faulty heater	Contact your local Thermo Fisher Scientific customer support organization.

## Low sensitivity.

Possible Cause	Possible Remedy								
Low photomultiplier excitation voltage	Increase the photomultiplier voltage.								
Inadequate hydrogen flow rate	Set the hydrogen flow rate to a proper value.								
Inadequate air flow rate	Set the air flow rate to a proper value.								
Dirty interferential filter	Clean the interferential filter.								
The mirror of the combustion chamber is dirty	Clean the mirror.								
The flame-side heat shield is dirty	Clean the flame-side heat shield.								
Reduced transparency of the heat shields	Replace the heat shields.								

## Low sensitivity: Water droplets generate between the heat shields.

Possible Cause	Possible Remedy
Leak at the flame-side heat shield	Replace the heat shields.
	If the symptom does not disappears, contact your local Thermo Fisher Scientific customer support organization.

# **PDD Analytical Troubleshooting**

If the system is clean, the signal value must be stabilized lower that 2000 pA. Observe the pink color of the discharge generated inside the detector. If a purple color of the discharge is observed, impurities or leaks in the discharge gas line are present.

#### **High Background Current**

When the background current is high check the discharge color is **pink** or **purple**.

#### The discharge color is pink.

Disconnect the column and cap the column inlet.

• The baseline current falls to normal range <2 nA.

The detector is not leaking. Check for the column bleed, leaks in the GC setup, or both.

Verify that the baseline current falls to normal range. If not, contact your local Thermo Fisher Scientific customer support organization.

• The baseline is still high >2 nA.

Perform a hydrogen leak test. See "Performing a Hydrogen Leak Test" on page 340.

- If leaks are present in the fittings of the PDD plumbing, tighten the leaking fittings.
   Verify that the baseline current falls to normal range.
- If Leaks are present inside the detector cell, contact your local Thermo Fisher Scientific customer support organization.
- If no detectable leaks are present, the carrier gas is possibly bad or the helium cylinder is empty. Install a new cylinder.
   Verify that the baseline current falls to normal range. If not, contact your local

Thermo Fisher Scientific customer support organization.

#### The discharge color is purple.

• The baseline is high >2 nA.

Perform a hydrogen leak test. See "Performing a Hydrogen Leak Test" on page 340.

- If leaks are present in the fittings of the PDD plumbing, tighten the leaking fittings.
   Verify that the baseline current falls to normal range.
- If Leaks are present inside the detector cell, contact your local Thermo Fisher Scientific customer support organization.
- If no detectable leaks are present, the carrier gas is possibly bad or the helium cylinder is empty. Install a new cylinder.

Verify that the baseline current falls to normal range. If not, contact your local Thermo Fisher Scientific customer support organization.

#### Low Sensitivity

When the detector sensitivity is low, check the background current with the column removed and with the column inlet plugged.

#### Background current is high (>2 nA)

See the troubleshooting "High Background Current" on page 338.

#### Background current is normal (<2 nA)

Reinstall the column and check the background current increases or remains in normal range.

• The background current increases.

Check column inlet for leaks. If leaks are present, tighten the leaking fittings.

- If the background current remain high, remove the column and condition it, then run a sample. If the sensitivity is still too low, check the position of the column and run a sample again.
- If the background current drops to normal range, run a sample.
   If the sensitivity is still too low, check the position of the column and run a sample again.
- The background current remain in normal range.

Check the position of the column and run a sample.

If the sensitivity is still too low, remove the column and bake the detector at 300 °C for four hours, then reinstall the column and run a sample.
 If the sensitivity is still too low, contact your local Thermo Fisher Scientific customer support organization.

#### **No Peaks**

If the background current is stable but there are not peaks, proceed as follows

- 1. Check the column flow.
- 2. Check the column insertion position.
- 3. If there are still no peaks, make sure that an analysis is running. If there are still no peaks, contact your local Thermo Fisher Scientific customer support organization.

#### **High Noise Level**

If the noise level is high, proceed as follows.

1. Check the column insertion position.

- 2. If the noise is still high, perform a hydrogen leak test. See "Performing a Hydrogen Leak Test" on page 340.
- 3. If there are no leaks found, or leaks are found and repaired and the noise level is still high, contact your local Thermo Fisher Scientific customer support organization.

#### **Performing a Hydrogen Leak Test**

A hydrogen leak test allows you to pinpoint loose fittings and leaks inside the detector.

- 1. Connect a length of plastic tubing to a regulated hydrogen bottle.Establish a flow of 5-10 mL/min.
- 2. Hold the hydrogen outlet tube at a fitting connection for ten seconds while monitoring the baseline.
- 3. Tighten the fitting and test it again, repeating as necessary until every connection has passed the ten second test.
- 4. Hold the hydrogen outlet close to the detector. (Do not insert the tube into any detector holes.) If a leak in the detector is indicated, contact your local Thermo Fisher Scientific customer support organization.

# **GDI Analytical Troubleshooting**

Refer to the troubleshooting guide of the third-party detector installed into the GDI mechanical module of the Generic Detector Interface.

# Glossary

This section lists and defines terms used in this guide. It also includes acronyms, metric prefixes, and symbols.

Α	B	С	D	E	F	G	H	1	J	K	L,	Μ	Ν	0	Ρ	0	R	S	Т	V	V	W	X	Y	Ζ	

#### A

A ampere

ac alternating current

ADC analog-to-digital converter

AOI Analog Output Interface

#### В

**b** bit

**B** byte (8 b)

**baud rate** data transmission speed in events per second

#### C

C Carbon °C Celsius CDS Chromatography Data System CIP Carriage and Insurance Paid To cm centimeter

 $\label{eq:cpu} \textbf{CPU} \ \ \textbf{central} \ \textbf{processing unit} \ (\textbf{of a computer})$ 

<Ctrl> control key of the keyboard

G

#### D

d depth DAC digital-to-analog converter dc direct current DS data system E ECD Electron Capture Detector EMC electromagnetic compatibility ESD electrostatic discharge F f femto oF Fahrenheit

FID Flame Ionization Detector

FOB Free on board

FSE Field Service Engineer

FPD Flame Photometric Detector

#### Glossary: ft

#### ft foot

**FT-IR** fourier transform infrared spectroscopy/fourier transform infrared spectrometer

#### G

**g** gram

GC gas chromatography- gas chromatograph

GDI Generic Detector Interface

**GND** electrical ground

**GSV** Gas Sampling Valve

#### Η

**h** height

 $\boldsymbol{h} \ \text{hour}$ 

H Hydrogen

**harmonic distortion** A high-frequency disturbance that appears as distortion of the fundamental sine wave

He Helium

HeS-S/SL Instant Connect Helium Saver Injector

HV high voltage

Hz hertz (cycles per second)

#### I

ID inside diameter

IEC International Electrotechnical Commission

#### Impulse See transient

in. inch

I/O input/output

#### K

**k** kilo  $(10^3 \text{ or } 1024)$ 

- K Kelvin
- kg kilogram

kPa kilopascal

#### L

*l* length

L liter

LAN Local Area Network

lb pound

LED light-emitting diode

#### Μ

m meter (or milli [10<sup>-3</sup>])
M mega (10<sup>6</sup>)
μ micro (10<sup>-6</sup>)
MBq megabecquerel
Ci millicurie
min minute

mL or ml milliliter

**mm** millimeter

MS mass spectrometry-mass spectrometer

m/z mass-to- charge ratio

#### Ν

**n** nano (10<sup>-9</sup>)

N Nitrogen

**negative polarity** The inverse of a detector signal polarity.

**nm** nanometer

NPD Nitrogen Phosphorous Detector

#### 0

OD outside diameter

 $\Omega$  ohm

#### Ρ

**p** pico  $(10^{-12})$ 

Pa pascal

PCB printed circuit board

PDD Pulsed Discharge Detector

**PN** part number

psi pounds per square inch

PTV Programmable Temperature Vaporizing Injector

**PTVBKF** Programmable Temperature Vaporizing Injector with backflush

#### R

RAM random access memory

<Return> <Return> key on the keyboard

RF radio frequency

**ROM** read-only memory

RS-232 industry standard for serial communication

#### S

s second

sag See surge

**slow average** A gradual long-term change in average RMS voltage level, with typical duration greater than 2 s.

SOP Standard Operating Procedures

SSL split/splitless injector

SSLBKF split/splitless injector with Backflush

**source current** The current needed to ignite a source, such as a detector lamp.

**surge** A sudden change in average RMS voltage level, with typical duration between 50 µs and 2 s.

#### Т

TCD Thermal Conductivity Detector

**transient** A brief voltage surge of up to several thousand volts, with a duration of less than 50 µs.

#### V

 $V \ \text{volt}$ 

Vac volts, alternating current

Vdc volts, direct current

VGA Video Graphics Array

#### W

 $\boldsymbol{w}$  width

 $\mathbf{W}$  Watt

When a unit of measure has a quotient (e.g. Celsius degrees per minute or grams per liter) this can be written as negative exponent instead of the denominator:

For example: °C min<sup>-1</sup> instead of °C/min g L<sup>-1</sup> instead of g/L

Glossary: W