



**Thermo Scientific** 

# **TRACE 1600/1610**

# **User Guide**

Gas Chromatographs

MI-317000-0034 Revision B January 2023



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Published by Thermo Fisher Scientific S.p.A., Strada Rivoltana, km 4, 20090 Rodano - Milan - Italy Tel: +39 02 950591

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Release history:

First Edition, released April 2022- "Original Instructions" Second Edition, released January 2023

General Lab Equipment. Not for Clinical, Patient, or Diagnostic Use.

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## **Reader's Survey**

#### TRACE 1600/1610 User Guide, MI-317000-0034, Revision B

	Strongly Agree	Agree	Neutral	Disagree	Strongly Disagree
The manual is well organized.	1	2	3	4	5
The manual is clearly written.	1	2	3	4	5
The manual contains all the information I need.	1	2	3	4	5
The instructions are easy to follow.	1	2	3	4	5
The instructions are complete.	1	2	3	4	5
The technical information is easy to understand.	1	2	3	4	5
Examples of operation are clear and useful.	1	2	3	4	5
The figures are helpful.	1	2	3	4	5
I was able to operate the system using this manual.	1	2	3	4	5

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Other\_\_\_\_\_

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# Declaration

#### Manufacturer: Thermo Fisher Scientific

Thermo Fisher Scientific is the manufacturer of the instrument described in this manual and, as such, is responsible for the instrument safety, reliability and performance only if:

- installation
- re-calibration
- changes and repairs

have been carried out by authorized personnel and if:

- the local installation complies with local law regulations
- the instrument is used according to the instructions provided and if its operation is only entrusted to qualified trained personnel

Thermo Fisher Scientific is not liable for any damages derived from the non-compliance with the aforementioned recommendations.

#### Thermo Fisher Scientific S.p.A.

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# **Regulatory Compliance**

Thermo Fisher Scientific performs complete testing and evaluation of its products to ensure full compliance with applicable domestic and international regulations.

Thermo Fisher Scientific declares, under sole responsibility, that the product as originally delivered complies with the requirements of the following applicable European Directives and carries the CE marking accordingly:

- Low Voltage Directive:2014/35/EU
- EMC Directive:2014/30/EU
- RoHS Directive 2011/65/EU, including amending Delegate Directive 2015/863/EU

... and conforms with the following product standards:

#### Safety

This device complies with:

- IEC 61010-1:2010/AMD1:2016, IEC 61010-2-010:2019
- CAN/CSA C22.2 No. 61010-1
- EN 61010-1:2020, EN 61010-2-010:2015
- UL 61010-1

#### **Electromagnetic Compatibility**

This device complies with:

- CISPR 11/EN 55011: Group 1 Class A
- IEC 61326-1:2012
- EN 61326-1:2012
- FCC part 15, Subpart B, §15.107(a) and §15.109(a)



**IMPORTANT:** Class A equipment is intended for use in an industrial environment. In others environments there may be potential difficulties in ensuring electromagnetic compatibility, due to the conducted as well as radiated disturbances.

# **FCC Compliance Statement**

THIS DEVICE COMPLIES WITH PART 15 OF THE FCC RULES. OPERATION IS SUBJECT TO THE FOLLOWING TWO CONDITIONS: (1) THIS DEVICE MAY NOT CAUSE HARMFUL INTERFERENCE, AND (2) THIS DEVICE MUST ACCEPT ANY INTERFERENCE RECEIVED, INCLUDING INTERFERENCE THAT MAY CAUSE UNDESIRED OPERATION.



**CAUTION** Read and understand the various precautionary notes, signs, and symbols contained inside this manual pertaining to the safe use and operation of this product before using the device.

# Notice on Lifting and Handling of Thermo Scientific Instruments

For your safety, and in compliance with international regulations, the physical handling of this Thermo Fisher Scientific instrument *requires a team effort* to lift and/or move the instrument. This instrument is too heavy and/ or bulky for one person alone to handle safely.

# Notice on the Proper Use of Thermo Scientific Instruments

In compliance with international regulations: Use of this instrument in a manner not specified by Thermo Fisher Scientific could impair any protection provided by the instrument.

# Notice on the Susceptibility to Electromagnetic Transmissions

Do not use radio frequency transmitters, such as mobile phones, in close proximity to the instrument.

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#### **Declaration of Conformity**

-Origina**l-**

CE

EU Declaration of Conformity



Thermo Fisher Scientific S.p.A. Strada Rivoltana 20053 Rodano Milan Italy

We hereby declare that the following products

Designation:

Model:

Thermo Scientific Trace 1600 Series Trace 1600, Trace 1610

2011/65/EU and (EU) 2015/863

EN 61326-1:2013-07

Gas chromatograph

2014/35/EU

fulfill all the relevant requirements of the following directives:

Low Voltage Directive

Electromagnetic Compatibility Directive 2014/30/EU

**RoHS** Directive

The following relevant harmonized standards were used:

EN 61010-1:2020-03

Person authorized to compile the technical file:

Giacinto Zilioli (Director, Strategic Projects) Thermo Fisher Scientific S.p.A.

Fracisto Milioli Signature

Milan, March 9, 2022

**UK Declaration of Conformity** 



-Origina**l-**

# Thermo Fisher

Thermo Fisher Scientific S.p.A. Strada Rivoltana 20053 Rodano Milan Italy

Declares, under sole responsibility, that products

Designation: Gas chromatograph

Model:

Thermo Scientific Trace 1600 Series Trace 1600, Trace 1610

as originally delivered complies with the essential requirements of the following applicable UK Regulations:

Electrical Equipment (Safety) 2016 Regulations

Electromagnetic Compatibility 2016 Regulations

The Restriction of the Use of Certain 2012 Hazardous Substances in Electrical and Electronic Equipment (ROHS) Regulations

and complies with the following harmonized standards and other technical specifications:

BS EN 61010-1:2010+A1:2019 BS EN 61326-1:2021

Signed for and on behalf of: Thermo Fisher Scientific S.p.A.:

Giacinto Zilioli (Director, Strategic Projects) Thermo Fisher Scientific S.p.A.

Signature

Milan, March 4, 2022 Date

## **China EEP Hazardous Substances Information**

产品中有害物质的名称及含量 China EEP Hazardous Substances Information

御件名称.	有害物质 Hazardous Substances   (TRACE 1600 Series)						
Component Name	∰ (РЬ)	汞 (Hg)	(Cd)	. <b>六价</b> 稿 (Cr(VI))	<b>多溴</b> 联苯 (PBB)	多溴二汞醚 (PBDE)	
(主机:習解接线电路板 ) Base Unit: PCBA BACKPLANE	х	0	0	0	0	0	
(主机:炉箱电路板) Base Unit: PCBA OVEN CPU	х	0	0	0	0	0	
(主机: 主控电路板) Base Unit: PCBA CPU	X	0	0	0	0	0	
(主机:存储器电路板) Base Unit: PCBA MEMORY	х	0	0	0	0	0	
(主机:楼口电路板) Base Unit: PCBA EXTERNAL INTERFACE	x	0	0	0	0	0	
(主机:电源供应电路板) Base Unit: PCBA POWER SUPPLY	x	0	0	0	0	0	
(主机:显示屏控制电路板) Base Unit: PCBA RSR1277	x	0	0	0	0	0	
(主机:USB接口控制电路板) Base Unit: PCBA RSR1284	x	0	0	0	0	0	
(主机:液晶显示屏) Base Unit: DISPLAY LCD 7"	x	0	0	0	0	0	
(分元/不分元进样器电路板) PCBA's MODULE SSL	x	0	0	0	0	0	
(現序升濃进祥器电路板) PCBA's MODULE PTV	x	0	0	0	0	0	
(火焰宮子伦恰测器电路板) PCBA's MODULE FID	x	0	0	0	0	0	
(电导检测器电路板) PCBA's MODULE ECD	x	0	0	0	0	0	
(復餐检测器电路板) PCBA's MODULE NPD	x	0	0	0	0	0	
(热导检测器电路板) PCBA's MODULE TCD	x	0	0	0	0	0	
(火焰光度检测器电路板) PCBA's MODULE FPD	x	0	0	0	0	0	
(辅助温度模块电路板)PCBA's MODULE AUXILIARY TEMPERATU	x	0	0	0	0	0	
(辅助气体模块电路板) PCBA MODULE AUXILIARY GASES	x	0	0	0	0	0	
(機似输出接口电路板) PCBA MODULE AOI	x	0	0	0	0	0	
(融冲放电检测器电路板) PCBA's MODULE PDD	X	0	0	0	0	0	
(通用检测器接口电路板) PCBA MODULE GDI	х	0	0	0	0	0	
(辅助炉箱电路板) PCBA's AUXILIARY OVEN	х	0	0	0	0	0	
(机加工件) MACHINED PARTS	0	0	0	0	0	0	
( <b>換只</b> ) MOLDED PARTS	0	0	0	0	0	0	
(概查件) SHEETMETAL PARTS	0	0	0	0	0	0	
(电机组件) ELECTROMECHANICAL ASSEMBLIES	0	0	0	0	0	0	
(电缆组件) CABLE ASSEMBLIES	0	0	0	0	0	0	
(标签) LABELS	0	0	0	0	0	0	

本最格依据SJ/T11364的规定编制 This table is compiled according to SJ/T 11364 standard.

O: 表示该有害物质在该部件所有均质材料中的含量均在GB/T26572规定的限量要求以下.

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这些产品的环保使用期为

The Environment Friendly Use Period for these products is:



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## WEEE Directive 2012/19/EU

This product is required to comply with the European Union's Waste Electrical & Electronic Equipment (WEEE) Directive 2012/19/EU. It is marked with the following symbol:



This symbol indicates that the equipment must not be thrown into general waste and should be collected separately and processed in accordance with local and state requirements.

Thermo Fisher Scientific is registered with one or more recycling/disposal companies in the UK and all other countries of the European Union and in Norway. If this product is located in Europe and you want to participate in the Thermo Fisher Scientific Business-to-Business (B2B) Recycling Program, send an email request to weee.recycle@thermofisher.com with the following information:

- WEEE product class
- Name of the manufacturer or distributor (where you purchased the product)
- Number of product pieces, and the estimated total weight and volume
- Pick-up address and contact person (include contact information)
- Appropriate pick-up time
- Declaration of decontamination, stating that all hazardous fluids or material have been removed from the product

For additional information about the Restriction on Hazardous Substances (RoHS) Directive for the European Union, search for RoHS on the Thermo Fisher Scientific European language websites.

**IMPORTANT** This recycling program is **not** for biological hazard products or for products that have been medically contaminated. You must treat these types of products as biohazard waste and dispose of them in accordance with your local regulations.

# Directive DEEE 2012/19/EU



Thermo Fisher Scientific s'est associé avec une ou plusieurs sociétés de recyclage dans chaque état membre de l'Union Européenne et ce produit devrait être collecté ou recyclé par celle(s)-ci. Pour davantage d'informations, rendez-vous sur la page www.thermoscientific.fr/rohs.



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# **Preface**

This guide contains detailed information for the use of the Thermo Scientific<sup>™</sup> TRACE<sup>™</sup> 1600/1610 GC. This guide is organized as follows:

#### **About Your System**

Thermo Scientific systems provide high-caliber gas chromatography (GC) instrumentation. Your TRACE 1600/1610 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<sup>™</sup> 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 1600/1610 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 Sheet.

## **Contacting Us**

There are several ways to contact Thermo Fisher Scientific for the information you need.

\* To find out more about our products

Go to http://www.thermofisher.com for information about our products.

#### ✤ To get local contact information for sales or service

Go to http://www.unitylabservice.com/en/home.html

#### **Related Documentation**

In addition to this guide, Thermo Scientific provides the following documents for the TRACE 1600/1610 gas chromatographs.

TRACE 1600 /1610 Document Set, P/N MI-317AH0-0038

- TRACE 1600/1610 Preinstallation Requirements Guide, P/N MI-317000-0032
- TRACE 1600/1610 Hardware Manual, P/N MI-317000-0033
- TRACE 1600 /1610 User Guide, P/N MI-317000-0034
- TRACE 1600/1610 Spare Parts Guide, P/N MI-317000-0035

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 xxv) 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 xxv). 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
	<b>BIOHAZARD:</b> 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.
	<b>ELECTRICAL SHOCK HAZARD:</b> Indicates that an electrical shock <i>could</i> or <i>might</i> occur.
	<b>FIRE HAZARD:</b> 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.
LUMMAR 2	<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.
	<b>MATERIAL AND EYE HAZARD.</b> Indicates you must wear eye protection when performing a task.
	<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.
<b>\$</b>	<b>LIFTING HAZARD</b> . Indicates that a physical injury <i>could</i> or <i>might</i> occur if two or more people do not lift an object.
	<b>MATERIAL AND EYE HAZARD:</b> Indicates that eye damage <i>could</i> or <i>might</i> occur.
2	<b>READ MANUAL:</b> 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 <i>could</i> or <i>might</i> put you at risk for a physical injury.
	<b>TOXIC SUBSTANCES HAZARD:</b> Indicates that exposure to a toxic substance could occur and that exposure <i>could</i> or <i>might</i> cause personal injury or death.
	<b>LASER HAZARD.</b> Indicates that exposure to a laser beam <i>will, could,</i> or <i>might</i> 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**

Table 1 explains the symbols used on Thermo Scientific instruments. Only a few of them are used on the TRACE 1600/1610, which are annotated with an asterisk below.

<b>Table 1.</b> Instrument Marking and Symbols (Sheet 1 of 2	Table 1.	Instrument Marking and Symb	ools (Sheet 1 of 2	)
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	Symbol	Description
		Direct current
*	$\sim$	Alternating current
	$\sim$	Both direct and alternating current
	3~~	Three-phase alternating current
		Earth (ground) terminal
		Protective conductor terminal
	$\rightarrow$	Frame or chassis terminal
	Å	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

	Symbol	Description
*		Caution, hot surface
*		Caution, biohazard
		In-position of a bistable push control
	$\square$	Out-position of a bistable push control
	<u>+</u> <u>)-</u>	Jack socket
*		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.

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

#### **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 1600/1610

The use of hydrogen as a carrier gas, or as fuel gas for certain flame detectors, requires 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. Because of the risk of explosion, operators must take special precautions. Using hydrogen as a carrier gas requires the operator's extreme caution unless an SSL equipped with a Helium Saver-H<sub>2</sub> Safer<sup>TM</sup> device or a hydrogen sensor is installed in the GC oven.

When using hydrogen as a carrier gas, you must equip your gas chromatograph with a hydrogen sensor or Helium Saver-H<sub>2</sub> Safer device.



Never use hydrogen as a carrier gas in your TRACE 1600/1610 system unless your oven has a hydrogen sensor installed or you equip the system with the Helium Saver-H<sub>2</sub> Safer device. Thermo Fisher Scientific FSEs are not authorized to install or repair any instrument using hydrogen as a carrier gas unless equipped with the appropriate sensor or the Helium Saver-H<sub>2</sub> Safer device.

Contact your Thermo Fisher Scientific sales representative if you do not have a Helium Saver- $H_2$  Safer or a hydrogen sensor installed in your oven. To comply with instrument safety requirements, a Thermo Fisher Scientific FSE or authorized service personnel should install the sensor into your TRACE 1600/1610.

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 in the case oven elements are not perfectly connected to each other, or when 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 1600/1610 has a Thermo Fisher Scientific hydrogen sensor or Helium Saver-H<sub>2</sub> Safer device 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 produce hydrogen in the quantity used in the GC.

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. The lower flammable limit of hydrogen in air is 4%.

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

**IMPORTANT** Use a hydrogen generator that uses a palladium dryer or a proton-exchange membrane to create hydrogen gas from deionized water. Use pressure swing adsorption and molecular sieve technology to remove moisture content down to trace levels.

• 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 Started with Your TRACE 1600/1610**

This chapter provides information to familiarize you with the TRACE 1600/1610 gas chromatographs.

#### Contents

- Instrument Basics
- Label Locations on the Instrument
- Oven
- Injector Modules Housing
- Detector Modules Housing
- External Modules Housing
- Gas Inlet Ports
- Electronic Module
- TRACE 1610 User Interface
- TRACE 1600 User Interface

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## **Instrument Basics**

The TRACE 1600/1610 contains the following key components:

- 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** such the as Auxiliary Temperature/Cryo, Auxiliary Gas, NPD thermionic source power supply, GDI Electrical Interface (part of the Generic Detector Interface), and Analog Output Interface modules.
- A touch screen **user interface** for TRACE 1610.
- A status panel **user interface** for TRACE 1600.



Figure 1. TRACE 1610 Gas Chromatograph with Touch Screen

Figure 2. TRACE 1600 Gas Chromatograph with Status Panel







**Note** The TRACE 1600/1610 can be furnished coupled with the TRACE 1600 Auxiliary Oven. See Figure 4.



The TRACE 1600 Auxiliary Oven is described in detail in the *TRACE 1600 Auxiliary Oven Instruction Manual* and as additional section of the *TRACE 1600 / 1610 User Guide* and *TRACE 1600 / 1610 Hardware Manual*.

## Label Locations on the Instrument

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



**Figure 5.** TRACE 1600/1610: Serial Number and US Patents Labels





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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.



#### Figure 10. Oven Internal View

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. Opening the door also switches on the oven light to facilitate user operations inside the oven.





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 12 and Figure 13 respectively.





Figure 13. 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 14.

Figure 14. Column Fitting Protruding



Fitting for the connection of the column

### iConnect Column Lock

The TRACE 1600 Series GC systems are equipped with instant connect (iConnect) Column Lock connectors that simplify capillary column installation, eliminating the risk of over tightening and assuring quick and leak-free operations.

You can make finger-tight connections in a simple click and capillary column length adjustments before installation. iConnect Column Lock connectors are compatible with iConnect SSL injectors and all iConnect detector modules, as well as with the Thermo Scientific TriPlus 500 Headspace Autosampler.



Figure 15. iConnect Column Lock



**CAUTION** The iConnect Column Lock has a maximum operating temperature of 350 °C. Do not use the iConnect Column Lock with an oven, inlet, or detector set to temperatures exceeding 350 °C.

**Note** For iConnect Column Lock installation instructions, refer to the *Installing a Column for the First Time* section of Chapter 1, in the *TRACE 1600/1610 Hardware Manual*.

#### **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 or TSQ Series 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 1600/1610 Hardware Manual*.

Figure 16 shows the position of the duct on the left wall through which an ISQ or TSQ Series transfer line inner tube will extend into the GC oven.



**Figure 16.** Duct for the Coupling with an ISQ or TSQ Series

You can also configure a stand-alone GC for coupling with an ISQ or TSQ Series mass spectrometer by installing the dedicated upgrade kit. For further details, please refer to the section *Upgrading a Stand Alone TRACE 1600/1610 to MS Version* in the *TRACE 1600/1610 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 17.





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 1600/1610 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 18.





Install the column rack by inserting the two hooks into the corresponding slots on the oven heater baffle. See Figure 19.



Figure 19. Column Rack (2)

For further details, please refer to the **Installation** section of the *TRACE 1600/1610 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 1600/1610 accommodates up to two injector modules in the **front/back** sites located on the **right** side. See Figure 21.

The housing must always be occupied by both the modules. According to the configuration of your TRACE 1600 or TRACE 1610 GC, a dummy module is present in the free site where the injector module is not installed.



Figure 21. Injector Housing View

Each front/back position has its own electrical and gas connections as shown in Figure 22.

Figure 22. 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 7, "Split/Splitless Injector (SSL) Module," on page 133
- Chapter 8, "Split/Splitless Backflush Injector (SSLBKF) Module," on page 149
- Chapter 9, "Gas Sampling Valve (GSV) Module," on page 163
- Chapter 10, "Instant Connect Helium Saver Injector Module (HeS-S/SL)," on page 175
- Chapter 12, "Programmable Temperature Vaporizing Injector (PTV) Module," on page 213
- Chapter 13, "Programmable Temperature Vaporizing Backflush Injector (PTVBKF) Module," on page 237

### **Detector Modules Housing**

Your TRACE 1600/1610 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 23. The housing must always be occupied by both the modules. According to the configuration of your TRACE 1600 or TRACE 1610 GC, a dummy module is present in the free site where the detector module is not installed.



Figure 23. Detector Housing View

Each front/back position has its own electrical and gas connections as shown in Figure 24.



Figure 24. 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 14, "Flame Ionization Detector (FID) Module," on page 255
- Chapter 15, "Nitrogen Phosphorus Detector (NPD) Module," on page 263
- Chapter 16, "Thermal Conductivity Detector (TCD) Module," on page 273
- Chapter 17, "Electron Capture Detector (ECD) Module," on page 285
- Chapter 18, "Flame Photometric Detector (FPD) Module," on page 295
- Chapter 19, "Instant Connect Pulsed Discharge Detector (PDD)," on page 309
- Chapter 20, "Generic Detector Interface (GDI)," on page 319

**Note** For details about the No Vent Microfluidics and FTIR Make-up **optional modules**, see the Chapter 22, "Optional Modules," on page 331.

### **External Modules Housing**

Your TRACE 1600/1610 can accommodate up to two external modules in the appropriate sites located in the back lower part of the GC. See Figure 25.





• 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 26.

#### Figure 26. 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 20, "Generic Detector Interface (GDI)," on page 319.
- 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 21, "Analog Output Interface (AOI) Module," on page 327.

### **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 27.





**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 1050 kPa (152 psig; 10.50 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 1050 kPa (152 psig; 10.50 bar).

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

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

**Table 1.** Capillary and Wide-Bore Column Gas Recommendations

**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 1600/1610. 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.

For precautions and use of hydrogen with the TRACE 1600/1610, see the section "Hydrogen Safety Precautions" on page xxviii for details.

#### **Using a Hydrogen Sensor**

The hydrogen sensor must be installed into the back of the GC as shown in Figure 28.



Figure 28. Hydrogen Sensor Housing

To install the hydrogen sensor into the GC, please refer to the *TRACE 1600/1610 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 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 29. Electronic Module



The Electronic Module (see Figure 29) 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 1600/1610 and the external devices such an autosampler or a computer. Figure 30 shows the components present on the external interface.



Figure 30. Electronic Module: External Interface

1. Two 6-pin connectors marked Front HSV and Back HSV.

**Note** When plugging in the 6-pin connectors make sure to align and fully insert the connectors until the retaining clips are securely engaged.

2. Two Mini Delta connectors marked **Front AS** and **Back AS** to connect to an autosampler on the front or back injector.

**Note** When plugging in the Mini Delta cables make sure to align and fully insert the connectors until the retaining clips are securely engaged.

- 3. A RJ45 connector marked **LAN/Ethernet** for the network connection of the TRACE 1600/1610.
- 4. A button marked **Reset IP** to reset the IP address.
- 5. A 6-pin connector marked **Autosampler Handshake** to synchronize the TRACE 1600/1610 with the autosampler.
- 6. An 8-pin connector marked **Generic Handshake** to synchronize the TRACE 1600/1610 with external devices.
- 7. Three 15-pin female connectors marked **Bus** for communication with external devices.

### **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 3 V; 220 mA/h non-rechargeable battery is present. A 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 1610 User Interface**

The TRACE 1610 user interface is a **touch screen** that provides immediate and detailed status displays, and guidance for ease of use and local control. See Figure 31.

Figure 31. TRACE 1610 User Interface



When the TRACE 1610 is powered on, the main menu appears as shown in Figure 32.





Each instrument function is associated with an icon that opens the a menu: Status, Maintenance, Signal, Instrument Control, Configuration, and Diagnostics.

See Chapter 3, "TRACE 1610 User Interface." for more details. To operate the TRACE 1600 through the Chromatography Data System, see Chapter 4, "Configuration and Method Parameters Setup from CDS."

## **TRACE 1600 User Interface**

The TRACE 1600 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 1600 gas chromatograph is the **status panel** shown in Figure 33. It contains three **light emitting diodes** (LED), showing the current instrument's status, and the **Start/Stop** and **Maintenance** function keys.



Figure 33. Status Panel

See Chapter 2, "TRACE 1600 User Interface." and Chapter 4, "Configuration and Method Parameters Setup from CDS." for details.

# **TRACE 1600 User Interface**

This section provides a description of the user interface of your TRACE 1600.

#### Contents

- TRACE 1600 User Interface Overview
- Configuring Your System and Method Parameter Setup

### **TRACE 1600 User Interface Overview**

The user interface of the TRACE 1600 gas chromatograph is the **status panel** shown in Figure 34. The panel has four **light emitting diodes** (LEDs), indicating the instrument's current status. There is also a **Start/Stop** and **Maintenance** function keys.



Figure 34. TRACE 1600 Status Panel

The following table describes TRACE 1600 LED states.

LED	State	Description
Power	Solid Green	Standby
Ready	Blinking Orange	Not ready
	Blinking Green	Preparing for analytical run.
	Solid Green	Ready to inject.
Run	Solid Blue	Analytical run in progress.
	Off	Cooling process
Maintenance	Blinking Green	Cooling for maintenance.
	Solid Green	Ready for maintenance.
Power and Ready	Blinking and audible beep	Error detected. Power, temperatures, and gases are automatically cut off.
Power and Maintenance	Solid Green	Firmware update.
All LEDs	Solid	Instrument power up.

**Table 3.** TRACE 1600 Status Panel LED States

TRACE 1600 status panel LED states are described when performing the following tasks:

- Power on the GC.
  - All LEDs on the status panel light up simultaneously. Next, the Power light becomes a solid green while all the other lights turn off.
  - The GC goes to **stand-by** status. The **Power** light remains a solid green while all the other lights are off.
- Analysis
  - 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.

When the parameters are ready, the **Ready** light turns off, and the GC goes into **standby mode**. Only the **Power** light is on.

 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.

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

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.

**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.

 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.

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

• Maintenance

The maintenance function is not available during a run.

 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.

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.
- When the GC is ready for maintenance, the light turns solid green.
- When maintenance is completed, press the Maintenance key. The light on the key turns off while the Ready light blinks orange indicating not ready status.

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

- Error
  - 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.
- Firmware update
  - When upgrading embedded firmware, the Power and Maintenance lights are solid green, the Ready light is off, and the Run light is solid blue.

## **Configuring Your System and Method Parameter Setup**

The TRACE 1600 does no have a keyboard or display. It is fully controlled by a computer using a Thermo Scientific Chromatography Data System (CDS) 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,"

# **TRACE 1610 User Interface**

This chapter describes the user interface of the TRACE 1610.

#### Contents

- TRACE 1610 User Interface Overview
- Configuration
- Instrument Control
- Instrument Status
- Signal Levels
- Diagnostics
- Maintenance
- AI/AS 1610

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## **TRACE 1610 User Interface Overview**

The TRACE 1610 user interface is a **touch screen** that provides immediate and detailed status displays and guides you for ease of use and local control. When the TRACE 1610 is powered on, the touch screen main menu will display. See Figure 35.



Figure 35. Touch Screen Main Menu

#### **Menu Icons**

Each instrument function is associated with an icon that opens a menu:

- Press the **Status** icon to monitor the status of the instrument. See "Instrument Status" on page 59.
- Press the **Maintenance** icon when the maintenance of one or more components of your GC is required. See "Maintenance" on page 70.
- Press the **Signal** icon to display the signal level of the front and back detectors. See "Signal Levels" on page 64.
- Press the **Instrument Control** icon to program and view method parameters for Oven, Front Inlet, Back Inlets, Front Detector, Back Detectors, Auxiliary Zones, and valves. See "Instrument Control" on page 52.
- Press the **Configuration** icon to configure your system. See "Configuration" on page 37.
- Press the **Diagnostics** icon to view diagnostic information. See "Diagnostics" on page 65.

#### **Active/Inactive Icons**

Each menu displays icons that corresponds to a module or component.

When a module or component is installed on the GC system, the icon is blue and available to open. When a module or component is not installed on the GC system, the icon is gray indicating that the module or component is unaccessible.

Figure 36. Active and Inactive Icons



#### **Data Entry Keys**

A keyboard displays in the **Instrument Control** and **Status** menu's left pane when you press a parameter name or an **actual** or **setpoint** field.

The numeric keyboard includes numbers from 0–9, 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 37.





- Use the **Enter** key to confirm the entry or the change made.
- Use the 🙁 key to erase the last digit typed.
- Use the **On/Off** key to set the setpoint value to Off or On.
- Use the < key to close the keyboard.

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

1. Press the name of parameter to enter or modify, for example **Temperature**. The value in the **Setpoint** field highlights.

2. Type a value, for example **250**, and then press **Enter** to confirm the modification.

### **Shortcut Keys**

Shortcut keys are located on the status/message bar of each menu and submenu.

The shortcut keys are:



### **Page Keys**

These keys indicate when other menu items exist. They appear in three ways:

- **Back** key < To view the previous page.
- View More key 🔽 To view the next page.

### Please Wait...

The following icon indicates you should wait because the system is performing a task or a setting.
# Configuration

The TRACE 1610 is configured to your specifications at the factory. You might need to reconfigure the instrument when you make changes to components.

When adding new components or changing injector modules, detector modules, carrier gases, and column types, you must reconfigure the system.

Press the **Configuration** icon on the touch screen main menu to configure the TRACE 1610. See Figure 38.



Figure 38. Touch Screen Main Menu: Configuration

The Configuration menu will display. See Figure 39.



Press the MORE icon to access the next page of the Configuration menu when additional modules are installed. See Figure 40.



Figure 39. Configuration Menu - First and Second Page

Figure 40. Configuration Menu - Third Page



Press the an icon to open a configuration menu. See the following sections:

- "Oven Menu" on page 39
- "Back and Front Inlet Menu" on page 39
- "Handshaking Menu" on page 46
- "Back and Front Detector Menu" on page 42
- "Back and Front Column Menu" on page 45
- "Network Menu" on page 48
- "Time/Units Menu" on page 47
- "Touch Screen Menu" on page 47
- "Auxiliary Oven Menu" on page 49
- "Auxiliary Carrier Menu" on page 50

- "Aux Left and Right Detector Menus" on page 49
- "Auxiliary Temperature Menu" on page 50
- "Auxiliary Column Menu" on page 51
- "Analog Output Interface Menu" on page 45

### **Oven Menu**

**Oven** — Sets actions of the oven.

- Auto preparation run The choices are **On/Off**. Set **On** to turn on automatic Prep Run execution without pressing Prep Run.
- Auto start Turns on the automatic start execution without pressing the start key. The choices are **On/Off**.
- **Prep run timeout** [min] 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.
- Equilibration time [min] 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** [min] 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 temperature [°C] 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**

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

The TRACE 1610 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>, and Ar.
- Backflush Choose Enabled or Disabled.

#### **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** 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 PTV BKF 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 cleaning? 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,  $N_2$ , Ar/CH<sub>4</sub>, Ar, none.
- Backflush 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.

#### **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 and Front Detector Menu**

**Back/Front Detector** — Configures the detector module installed into the back/front position of the GC, and into the left/right auxiliary position of the TRACE 1600 Auxiliary Oven when present.

The TRACE 1600/1610 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  $N_2$  and  $Ar/5\%CH_4$ .

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 of 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  $\mathrm{N}_2$  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 1600/TRACE 1610 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 21.

- 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 1600 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 user interface. See **Run Time Event 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/Front Column — Evaluates the column in use on the back/front position.

Set the following parameters:

- **Description** Enter a description of the column.
- **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** If a precolumn is present select **On**, otherwise select **Off**. 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 as 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** If a post column is present select **On**, otherwise select **Off**. 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 as 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.
- **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 accuracy for the column carrier flow rate calculation, the Column Evaluation procedure is recommended.

• **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 1600/1610 Hardware Manual* for more details.

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.

### **Handshaking Menu**

**Handshaking** — Configures 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 (In)** Allows another device to start the GC. The choices are **L to H** (low to high) or **H to L** (high to low).
- Inhibit ready (In) Delays readiness until the GC receives a signal from another device. The choices are L (low), H (high), or None.
- 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).
- 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).
- **Ready (Out)** Signals another device that the GC is ready. The choices are **Low** or **High.**
- **Preperation run (Out)** Signals another device that the GC is preparing for a run. The choices are **Low** or **High**.

Default setting for the Thermo Scientific<sup>™</sup> autosamplers are:

Remote Start: H to L	Inhibit Ready: None	End of Run: H to L	Start of Run: H to L
Ready Out: Low	Prep Run: Low	Remote Start should be	e set Active Low

### Time/Units Menu

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

- **Date** Enter the date (mm.dd.yyyy) 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 units Choose the pressure unit among psi, kPa and bar.

### **Touch Screen Menu**

**Touch Screen** — Press this icon to choose a language, adjust brightness and volume, set the screen saver, and calibrate the display. See Figure 41.



Figure 41. Touch Screen Menu

- Language English is set as the default language. Choose an available language. Once a language is selected, menus and all other windows, functions, and messages will display in that language.
- **Brightness** Adjusts the brightness of the screen. Use the left/right arrows to decrease/increase the brightness level.
- **Volume** Adjust the volume of the sound heard when a icon is pressed. Use the left/right arrows to decrease/increase the volume level.
- Main menu screen saver Blanks the images on the screen after a period of inactivity. Select On or Off. When on the function is enabled; when off the function is disabled.

• **Display calibration** — Press this button to calibrate the touch screen. A cross appears in the center of the page. Press the cross with a pen or with a finger of your hand; continue to press the cross according to the instructions 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 42.



Figure 42. Network Menu

The TRACE 1610 ships 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 an IP address to be assigned, the netmask, and eventually the port.

- GC name is the name you assign the GC.
- The **IP** Address is a 3 digits x 4 fields given by the network administrator, e.g. 192.168.127.10.
- The **Subnet Mask** is a 3 digits x 4 fields given by the network administrator e.g. 255.255.255.0
- Gateway is the IP Address of the default gateway.
- DHCP Enabled list if Dynamic Host Configuration Protocol (DHCP) is enabled.
- The MAC field list the MAC address.

When the parameters are set, press Apply.



**CAUTION** The GC will reboot when you apply the new network settings.

### **Auxiliary Oven Menu**

Use this menu to configure the heating zones of the TRACE 1600 Auxiliary Oven. See Figure 43.

Figure 43. Auxiliary Oven Menu

Heater 1:	Valve Oven	$\sim$
Heater 2:	Aux 4	~
Valve number	1	~
Valve type		~

- 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.
- Valve number: Select a valve number in the range of 1-8.
- Valve type: Select Sampling or Switching according to the valve type.

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

### **Aux Left and Right Detector Menus**

**Aux L Det/Aux R Det** — Configures the auxiliary detector module installed into the left/right position on the TRACE 1600 Auxiliary Oven (Valve Oven).

The configuration menu is the same as the detector modules installed in front and back positions on the GC. See "Back and Front Detector Menu" on page 42 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 44.

Figure 44. Auxiliary Carrier Gas Menu

Carrier Line	Aux 4	~
Gas type	Не	~

- **Carrier line** Select the auxiliary carrier line **Aux 1-6**.
- **Gas type** Select the carrier gas in use for the selected auxiliary carrier line. The choices are He, H<sub>2</sub>, and N<sub>2</sub>.

### **Auxiliary Temperature Menu**

**Auxiliary Temperature** — Configures the heater zones of the Auxiliary Temperature/Cryo module. See Figure 45.

Figure 45. Auxiliary Temperature Menu

Heater 1:	Aux 1	~
Heater 2:	X-line 2	~

- 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 46.

Carrier Line	Aux 4	N 1	1	Pre column?	No 🗸
Position	GC Oven		-	Pre column length [m]	0.00
Description	123456789	-12345678	9	Pre column ID [mm]	0.000
Column length [m]		0.00		Post column?	No 🗸
Column ID [mm]		0.000		Post column length [m]	0.00
Film thickness [um]		0.00		Post column ID [mm]	0.000
Page	e1of2	VIEW MORE	•	BACK Page 2 of 2	

#### Figure 46. Auxiliary Column Menu

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

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.
- **Column length (m)** Enter the length of the column.
- Column ID (mm) Enter the internal diameter of the column.
- Film thickness  $(\mu m)$  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.

# **Instrument Control**

This section describes how you can program the control parameters for all 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 to program the parameters in the analytical method. See Figure 47.

Figure 47. Touch Screen Main Menu: Instrument Control



The Instrument Control menu displays. See Figure 48.

Figure 48. Instrument Control Menu



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

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



**CAUTION** Parameters cannot be programmed while an analysis is running.

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

### **Oven Parameters**

**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 detailed descriptions see Using Oven Parameters section in Chapter 6.

### **Back and Front Inlet Parameters**

**Back and 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 7.
- Split/Splitless Injector for Backflush applications (SSLBKF) See Using SSLBKF Parameters in Chapter 8.
- Gas Sampling Valve (GSV) See Using GSV Parameters in Chapter 9.
- Helium Saver (HeS S/SL) See Using Instant Connect Helium Saver Injector Module Parameters in Chapter 10.
- Programmable Temperature Vaporizing Injector (PTV) See Using PTV Parameters in Chapter 12.
- Programmable Temperature Vaporizing Injector for Backflush application (PTVBKF). See Using PTVBKF Parameters in Chapter 13.

# **Back and Front Detector Parameters**

**Back and 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 14.
- Nitrogen Phosphorus Detector (NPD) See Using NPD Parameters in Chapter 15.
- Thermal Conductivity Detector (TCD) See Using TCD Parameters in Chapter 16.
- Electron Capture Detector (ECD) See Using ECD Parameters in Chapter 17.
- Flame Photometric Detector (FPD) See Using FPD Parameters in Chapter 18.
- Pulsed Discharge Detector (PDD) See Using PDD Parameters in Chapter 19.
- Generic Detector Interface (GDI) See Using GDI Parameters in Chapter 20.

### **Aux Left and Right Detector Parameters**

**Aux L Det and Aux R Det** — This section lists the parameters for programming the detector modules installed into the left/right position on the TRACE 1600 Auxiliary Oven (Valve Oven). The parameter 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 54 for details.

## **Run Time Event**

**Run Time Event**— Set detectors, valves, 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. See Figure 49.

The timetable has three columns: Time, Item, and Action. 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 using the Add, Modify, and Remove buttons.

The Add button opens the **Run-Time Event** screen. See Figure 49.

Time:	Prep-run	~		Time:	Prep-run	~	
Item:	Detector	~		Item:	Aux Gas	✓ # 2	~
	FID-Front	~					
Action:	Autozero	~		Action:	Off	$\sim$	
	ОК	Cancel			ОК	Cancel	
		Time:	Run time	~	10.00		
		Item:	Detector	~			
			FID-Front	~			
		Action:	Flame Off	~			
			OK	Can	cel		
			OK	Call			

Figure 49. 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 action 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<sup>0</sup> to 10<sup>3</sup> of the selected **Channel** at the given time.

### **Auxiliary Temperatures**

**Auxiliary Temp.** — Defines temperature parameters for the external auxiliary temperature modules set in the configuration. See Figure 50.

Figure 50. Auxiliary lempe	erature Menu
----------------------------	--------------

Instrument control - Auxiliary Temp.		07 Apr 2021	16:30 <b>25 °C</b>		
	( <u>†</u>	<b>†</b>		Actual	Setpoint
			Aux 1	24	Off
BACK	Aux L Det. VVV	Aux R Det. xxx	Aux 2	24	Off
		$\bigcirc$			
Auxiliary Temp.	Aux Oven	Aux. Carrier			
♠ ♥	ш Ш	Ready	to Inject	Start	Stop

- Aux 1— Enter the Setpoint for Aux 1.
- Aux 2— Enter the Setpoint for Aux 2.

### **Events**

**Events** — Set events On or Off for up to eight external valves connected to the Aux Temperature/Cryo Module. See Figure 51.

**Instrument control - Events** 07 Apr 2021 17:12 25 °C t Event #1 default Off BACK Event #2 default vvv xxx Off Event #3 default Off Auxiliary Aux Oven Aux. Carrier Event #4 default On Temp. Event #5 default On  $\bigcirc$ Event #6 default On Events **VIEW MORE** M  $\checkmark$ Шп Start Stop

A second Valve page is available to set **On** or **Off** up to eight valves installed into the Valve Oven (TRACE 1600 Auxiliary Oven) when present.

# Aux Oven

**Aux Oven** — Sets the auxiliary temperatures of the TRACE 1600 Auxiliary Oven. See Figure 52.

#### Instrument control - Aux Oven 07 Apr 2021 17:44 25 °C Actual Setpoint ╈ Valve Oven 50 васк xxx 50 Aux Col. Oven $\bigcirc$ Ø Auxiliary Aux Oven Aux. Carrier Temp. VIEW MORE M ſh Start Stop $\checkmark$

**Valve Oven** — Enter the Setpoint for the Valve Oven.

Aux Col. Oven — Enter the Setpoint for Aux Col. Oven.

Figure 51. Events Menu

Figure 52. Aux Oven Menu

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

#### Table 4. 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** — Set up to six auxiliary carrier lines selected by the **Carrier Line** drop down menu. See Figure 53.





- **Carrier line** Select the auxiliary carrier line **Aux 1-6**.
- Flow mode Defines the auxiliary carrier gas control mode to use: Constant Flow, Constant Pressure, Ramped Flow, Ramped Pressure, and Constant Linear Velocity. Each mode activates or deactivates the dedicated parameters.
- **Pressure** Defines the actual and setpoint pressure of the carrier gas. The range is On/Off; 0-1050 kPa (0-152 psi; 0-10.50 bar).

Column flow — Defines the carrier gas flow rate through the column. The range is 0.01–100 mL/min. This line is not editable when the Constant Pressure or Ramped Pressure mode is selected.

# **Instrument Status**

Press the **Status** icon on the touch screen main menu to view the status of the TRACE 1610. See Figure 54.

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



Figure 54. Touch Screen Main Menu: Status

The Status menu appears. See Figure 55.

### Figure 55. Status Menu



Press an icon to open the submenu. See the following sections:

- "Run Status" on page 60
- "Pressure Status" on page 62
- "Zone Temperature" on page 62
- "Waiting For" 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 1610 status LEDs display during each stage in a run. See Figure 56.

Status - Run S	tatus		09 Apr 2021 1	11:26 <b>25 °C</b>
く 🛛 Last run t	i <b>me</b> 5.67	Next run t	t <b>ime</b> 12.34	ł
Stage 1	Stage 2	Stage 3	Stage 4	Stage 5
Ready to Inject	Initial Temperature	Ramp	Final Temperature	Cooling
I ●	NOT READY O STAND	BY O RUN LOG	GAS SAVER	
🏠 🗞 मा	Read	dy to Inject	Start	Stop

Figure 56. Run Status

The oven ramp LEDs indicate temperature ramp stages during a run. You can follow the progress of a run by observing the LEDs and the message in the Status bar. See Figure 57.

Status – Run S	Status – Run Status			1:43 AM 25 °C
く Last run tim	n <b>e:</b> 12:08   <b>Next ru</b>	<b>un time:</b> 14:15		
Stage 1	Stage 2	Stage 3	Stage 4	Stage 5
Ready to inject	Initial Temperature	Ramp	Final Temperature	Cooling
🔍 RUN LOG 🔶 GA	S SAVER SOURCE SA	VER		
🔺 💙 📶		Ready		Start

Figure 57. Run Stages and Status LEDs

The stages in a run are:

- Not Ready The TRACE 1610 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 blue 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.
- 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 1610 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 69.

# **Pressure Status**

**Pressure Status** — Displays the current pressure of each injector module. The value is read only. See Figure 58.



Figure 58. Pressure Status

## **Zone Temperature**

**Zone Temperature** — Monitors the actual and setpoint temperature of all TRACE 1610 heated zones. You can modify the temperature setpoint of the zone of interest through the keyboard that appears on the left pane. See Figure 59.

Status - Zo	one tempe	rature		09	Apr 202	21 12:	36 <b>25 °C</b>
		<b>∩</b> ∎°C			Actual		Setpoint
$\bigcirc$		Zone	Oven [°C]		60	~	60
Pressure status	Run Status	Zone temperature	Back Inlet [°C]	SSL	25	~	Off
			Front Inlet [°C]	PTV	25	~	Off
Waiting for			Back Det. [°C]	TCD	27	~	Off
			Front Det. [°C]	FID	27	~	Off
	щ (W	Ready	to Inject 📃 🚺	Sta	art		Stop

Figure 59. Zone Temperature Status Page (1)

Figure 60. Zone Temperature Status Page (2)

Status - Zone temperature			C	09 Apr 2021 12:36 <b>25 °C</b>		
Pressure status	Run Status Zone tempera	<b>∩</b> ⊧°C		Actual		Setpoint
			Oven [°C]	25	0	60
		zone temperature	Back Inlet [°C] SSI	25	~	Off
			Front Inlet [°C] PT	25	~	Off
Waiting for			Back Det. [°C] TC	27	~	Off
			Front Det. [°C] FID	27	~	Off
	ъщ 🖤	Ready	to Inject 📃 下	Start		Stop

- A **green** background indicates the actual value has reached the programmed setpoint value.
- A **red** background indicates the actual value has not yet reached the programmed setpoint value.

# **Waiting For**

**Waiting for** — This function monitors and displays tasks or conditions the GC is waiting for to go to ready status. See the list in Figure 61.



Figure 61. Waiting For Function

# **Signal Levels**

Press the **Signal** icon on the touch screen main menu to monitor the signal level of the front and/or back detectors. See Figure 62.

Figure 62. Touch Screen Main Menu: Signal



The Signal page displays. See Figure 63.



Press the Front or Back tab to view the signal level of the detector.

# **Diagnostics**

Press the **Diagnostics** icon on the touch screen main menu to view diagnostic information. See Figure 64.



Figure 64. Touch Screen Main Menu: Diagnostics

The Diagnostics menu appears. See Figure 65.



Press an icon to open the submenu. See the following sections:

- "Software Info" on page 66
- "Hardware Info" on page 67
- "Network Info" on page 67
- "Readings" on page 68
- "Errors" on page 68
- "Run Log" on page 69
- "Save to USB" on page 70

# **Software Info**

**Software Info** — Lists the software or firmware version of the HMI, Main F/W, Main bootloader, Oven, and injector and detector modules. See Figure 66.



#### Figure 66. Software Info Page

## **Hardware Info**

**Hardware Info** — Lists the number of total number of runs performed, the manufacturing date, voltage, serial numbers for the components and modules installed, and  $H_2$  sensor information. See Figure 67.

Figure 67. Hardware Info Page



### **Network Info**

**Network Info** — Indicates the network connection specifications (IP address, Subnet mask, etc), and suffix DNS description. See Figure 68.



#### Figure 68. Network Info Page

# **Readings**

**Readings** — Displays the status of the various devices. See Figure 69.

#### **Figure 69.** Readings Page



### **Errors**

Errors— Lists any errors in device status codes. See Figure 70



#### Figure 70. Errors Page

# **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. See Figure 71

Figure 71. Run Log Page



# Save to USB

**Save to USB** — This command saves the contents of the Diagnostic sub-menus to a USB stick in text format. The filename is generated by the system and includes the name of the instrument, the date, and the time it was saved.

Figure 72. Save to USB Page



Figure 73. USB Port Location



# Maintenance

Press the **Maintenance** icon on the touch screen main menu to perform required maintenance. See Figure 74.

TRACE 1610		20 Jan 20	22 11:12 200 °C
Status	Maintenance	Signal	Instrument control
¢¢ Configuration	Diagnostics		
♠ ♥	Standby	► Prep.	

Figure 74. Touch Screen Main Menu: Maintenance

The Maintenance menu appears. See Figure 75.

### Figure 75. Maintenance Menu

Maintenance - Cool for maintenance 09 A					07 <b>25 °C</b>	
	*		Front (PTV) [°C]	Present Temperature 25	Cool this zone? Yes 🗸	
Counters	Cool for maintenance	View Logbook	Back (SSL) [°C]	25	Yes 🗸	
<b>I</b>	?		Front (FID) [°C]	27	Yes 🗸	
Leak Check	Quick reference	Video	Back (TCD) [°C]	27	Yes 🗸	
Select the zones you wish to cool for the maintenance activity then press the Begin Cooldown button			Restore temperatures			
♠ 💙	<b>M I</b> hr	Ready	to Inject 📃 🕨	Start	Stop	

Press an icon to open the a submenu. See the following sections:

- "Counters" on page 72
- "Cool for Maintenance" on page 73
- "View Logbook" on page 73
- "Leak Check" on page 74
- "Quick Reference" on page 74

• "Video" on page 75

## **Counters**

Select the **Counters** icon, or the **Diagnostics** icon on the taskbar, to access instrument health information and automated consumables tracking. The Counters page features information indicating when maintenance should be performed and when consumables are due for replacement. A red heart icon indicates an alert and green heart indicates good health.

Select each consumable to set an alert level according to the application, restore default values, or reset the counter to zero after replacement or performing maintenance. Use the keypad to enter the alert level. See Figure 76 and Figure 77.

Figure 76. Counters Page

Maintenance - Counters	09 Apr 2021 14:59 <b>25 °C</b>		
< Please check alerts for Front channel			
Front channel Back channel			
<u>Septum lifetime</u>	Injection 139 of 500		
Liner lifetime	Injection 408 of 500		
① <u>Column trimming</u>	Injection 1500 of 1500		
Inlet cleaning	Injection 11439 of 20000		
<u>Column replacement</u>	Injection 7450 of 20000		
Page 1 of 3	VIEW MORE		
🟫 💔 🏛 🛄 🦷 Ready to Inject	🕨 Start 🛛 🗖 Stop		

Figure 77. Setting Counter Alert Level

🔀 Trace 1610-TRACE 1610				×	
Maintenance - Counters		28 Ma	r 2022 17:42	170 °C	
	4	BACK			
NOTE: the alert level value is high dependent on the application. Tra	ly Face	Front channel - Septum lifetime			
value according to the application Please read the manual for more	. А	lert level	ł	5	
details.	S	Septum lifetime		2	
Set default value		Reset counter after mainter		ance	
🔺 🦁 🜆	Standby	► Pr	ep.		
## **Cool for Maintenance**

**Cool For Maintenance** — This function cools zones within the system for maintenance.

In this page, select **Yes** for the zones you wish cool down for maintenance activity, then press **Begin Cooldown**. See Figure 78.

Figure 78. Cool for Maintenance Page

Front (PTV) [°C]	Present Temperature 25	Cool this zone?				
Back (SSL) [°C]	25	Yes 🗸				
Front (FID) [°C]	27	Yes 🗸				
Back (TCD) [°C]	27	Yes 🗸				
Begin Cooldown						

## **View Logbook**

**View Logbook** — This function displays a logbook which records the time and a description of any maintenance performed. See Figure 79.

Figure 79. Maintenance - View Logbook Page

Maintenance	e - View Logbook	09 Apr 2021 14:	.32 <b>25 °C</b>			
Date & Time	Description					
apr/09/2021 14:22	Replaced septum (Front)					
apr/7/2021 10:50	Replaced liner (Front)					
apr/11/2021 11:39	Replaced Cleaned inlet (Front)					
apr/11/2021 16:48	Replaced Carrier filter (Front)					
apr/09/2021 14:22	Replaced septum (Back)					
apr/7/2021 10:50	Replaced liner (Back)					
apr/11/2021 11:39	Replaced Cleaned inlet (Back)					
apr/11/2021 16:48	Replaced Carrier filter (Back)					
Delete entr	y Delete all		Exit			
🏫 💙 🏛	🗊 🛄 Ready to Inject	Start	Stop			

**Delete entry** — Press this button to delete a single entry.

Delete all — Press this button to delete all logbook entries.

Exit — Press this button to exit the logbook.

## Leak Check

**Leak Check** — Use this function to perform a leak check. See Figure 80. This procedure requires a column-flowmeter connector. Refer to the *TRACE 1600/1610 Hardware Manual* for more details.

Figure 80. Leak Check Page

Maintenance - Column Leak check	09 Apr 2021 14:3	4 <b>25 °C</b>
	Inlet for leak check	Front 🗸
Counters Cool for View maintenance Logbook	Leak check pressure [psi]	0.0
	Allowed pressure drop [psi]	0.0
Leak Check Quick Video reference	Leak check time [min]	0.00
Select the inlet you wish to test for leaks. Plug the column exit for small leak detection of ferrules or septa. Alternatively,		
enter an allowed pressure drop for columns with open exits and known pressure drop characteristics.	Begin leak check	
🟫 💙 🏛 🛄 Ready to	) Inject 🕞 Start	Stop

In this page, select the front or back inlet, set the pressure and the duration of the leak check, and 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 81.

Maintenance - Quick reference		09 Apr 2021	14:35 <b>25 °C</b>
< SSL V			
Splitless: 5 mm He Saver: 5 mm	Split: I0 mm		
🟫 👽 🏛 퉲 🛛 Ready	to Inject	<b>&gt;</b> Start	Stop

Figure 81. Quick Reference Page

This page displays the column insertion depths for SSL, PTV, ECD, FPD, FID, NPD, TCD, GSV, PDD, and TSI.

Detector and injector insertion depths are measured from the top of the ferrule. To exit this page, press < button.

## Video

This page displays videos showing step by step instructions of related GC tasks and maintenance. The available videos are determined by the configuration of the instrument.

Figure 82. Video Page



# AI/AS 1610

Press the **AI/AS** icon on the touch screen main menu to view information and set. See Figure 83. **Figure 83**. AI/AS 1610



## Status

Press the Status icon to monitor the status information for the autosampler. See Figure 84.

**Sequence** — Displays the status of the sequence.

**Current vial** — Displays the vial number of the current vial.

Door — Displays the status of the door. Open or Closed.

Figure 84. AI/AS 1610 Status

Al/A	\S 16	10 - Status			04 Mar 2022 1	3:36	50 °C
	i		<b>†</b>	Sequence	Running	9	
St	tatus	Configuration	Instrument control	Current vial		2	
	20			Door	Closed		
h	nfo	Sampling	Maintenance				
					Running (Starting	)	
	V	₩.	Ready to	Inject	▶ Start	5	Stop

## Configuration

Press the **Configuration** icon to setup and configure an AI/AS 1610 sampling system. See Figure 85.

Syringe type ( $\mu$ L)— Choose the type of syringe: 0.5  $\mu$ L, 5  $\mu$ L, 10  $\mu$ L, 10  $\mu$ L (Teflon tip plunger), 50  $\mu$ L, 100  $\mu$ L, or Custom.

GC inlet mode — Choose the GC inlet mode: SSL, PTV, PTV\_OC, TSI, or Custom.

Injection depth (mm) — Enabled when Custom inlet mode is selected.

Penetration speed (mm/s)—Enabled when Custom inlet mode is selected.



Figure 85. AI/AS 1610 Configuration

## **Instrument Control**

Press the Instrument Control icon to program and view method parameters. Press Next to see more parameters. See Figure 86.

Injection type— The options are: Standard or Sandwich.

Sample mode — The options are: Custom, Standard, or Viscous.

Fill strokes — Determine how often the syringe plunger is moved up and down before the sample is actually drawn. This eliminates bubbles and thus, enhances reproducibility. The range is 0-15.

**Draw speed** — The draw speed is determined by the sample mode parameter. If **Custom** mode is selected, the draw speed is programmable. For Custom mode enter a draw speed between  $0.1-25 \mu l/s$ .

**Air volume** — Specifies the volume of air drawn after pulling the syringe needle out of the vial. This reduces evaporation from the syringe needle. The total of **Air Volume** plus **Volume** must not exceed the syringe volume. The range depends on the syringe volume.

**Sample depth** — Select how deep the needle descends into the vial when drawing the sample for injection. The options are: **Bottom** (33 mm) or **Half** (17.5 mm).

**Viscosity delay** — This parameter is enabled for **Viscous** sample mode. Set the delay after sample aspiration. Enter a value between 0 to 10 seconds.

Air gap 1—This parameter is enabled for Sandwich injections. Specify the gap between the two layers. Enter a volume based on the syringe selected.

Air gap 2—This parameter is enabled for Sandwich injections. Specify the gap between the first layer and the plunger. Enter a volume based on the syringe selected.

Layer 2 volume—This parameter is enabled for Sandwich injections. Set the volume of the layer closest to the plunger. Enter a volume based on the syringe selected.

Layer 2 source—Choose from a range of Solvent\_A to Sample\_155.

Figure 86. AI/AS 1610 Instrument Control

AI/AS 1610 - Instrum	ent control		04 M	ar 2022 13:3	7	50 °C
	the second se	Injection type		Standard		$\sim$
Status Configuration	Instrument	Sample mode		Standard		$\sim$
	control	Fill strokes			5	
		Draw speed [ul/	/s]		5.0	
Info Sampling	Maintenance	Air volume [ul]			0.0	
	Sample depth		Bottom		~	
		Viscosity delay	[s]		0	
			Page	1 of 5	ľ	Next 🕨
♠ ♥ 🜆	Ready to	Inject	•	Start	St	ор

### Info

Press the Info icon for reference information about your AI/AS 1610. See Figure 87.

AI/AS— Lists the autosampler configuration.

Serial Number— Displays the autosampler serial number.

**Firmware version**— Displays the installed firmware version.

Manufacturing date— Shows the date of manufacture.

AI/AS 1610 - Info 04 Mar 2022 13:37 50 °C AS 155 S/N: 000000000 (î Firmware Version: 00.38.12 Manufacturing date: 000000000 Status Configuration Instrument control Info Sampling Maintenance M Stop Start Ready to Inject ~

#### Figure 87. AI/AS 1610 Info

# Sampling

Press the Sampling icon to view sampler and injection settings. See Figure 88.

**Vial number**— Use the keypad to enter the vial number.

**Injection volume** ( $\mu$ L)— Use the keypad to enter the injection Volume.

Inject into— Pick where to inject into. The options are Injector or Waste.Figure 88. AI/AS 1610 Sampling

AI/AS 1610 - Sampling	04 Ma	ir 2022 13:38	50 °C
	Vial number	2	
Status Conliguration Instrument control	Injection volume [ul]	0.0	)
	Inject into	Injector	~
Info Sampling Maintenance	Start	Stop	
🟫 💙 🜆 🛛 Read	/ to Inject 💦 💦 🔊	itart 🧧 S	Stop

## Maintenance

Press the Maintenance icon to for washing and maintenance functions. See Figure 89.

Washing cycle pre— Select if there is a pre wash cycle. The options are Yes or No.

Washing cycle post— Select if there is a post wash cycle. The options are Yes or No.

**Start washing cycle** — Click this button to start a washing cycle.

**Stop washing cycle** — Click button to stop a washing cycle.

Figure 89. AI/AS 1610 Maintenance



# **Configuration Parameter Setup from CDS**

This chapter contains instructions to configure your TRACE 1600/TRACE 1610 and to edit the parameters (according to the front/back inlet, front/back/ detector modules, and optional devices installed), through Chromeleon or SII Thermo Scientific Chromatography Data System (CDS).

#### Contents

- General Configuration Tab
- Instrument Configuration Tab
- Oven Configuration Tab
- Inlet Configuration Tabs
- Detector Configuration
- Auxiliary Carrier Configuration Tab
- Valves & Events Configuration Tab
- Signals Configuration Tab
- Auxiliary Heaters Configuration Tab

**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.

4

# Introduction

Run your Chromatography Data System and open the TRACE 1600 Configuration window. See Figure 90.

	Figure 90	Configurati	ion Window	(Chromeleon	CDS
--	-----------	-------------	------------	-------------	-----

1000 M		00			_						
Device N	lame:	GC				E	essure Unit:	bar	~		
Network	Address:	192	168.127.10	č.		<u>C</u> r	yo:	None	<u> </u>		
ICP Por	t	255	1		÷		H2 Sensor Insta	alled			
Timeout		5			÷ s						
Device N	Aodel:	TR	ACE 1600/	1610	~	Li	e Frequency:	50	~		
Simulatio	on Mode:	0	Off OF	lead (	) <u>W</u> rite	e Se	erial Number:		H	z	
Simulatio	on Eile:	Tra	ce1600De	mo	~	Fi	mware Version:				
Lock	the GC fro	int panel									

The configuration window is subdivided into the following tabs.

- "General Configuration Tab" on page 83
- "Instrument Configuration Tab" on page 84
- "Oven Configuration Tab" on page 84
- "Front Inlet Configuration Tab" on page 85
- "Back Inlet Configuration Tab" on page 86
- "Front Detector Configuration Tab" on page 86
- "Back Detector Configuration Tab" on page 87
- "Auxiliary Carrier Configuration Tab" on page 88
- "Valves & Events Configuration Tab" on page 89
- "Signals Configuration Tab" on page 89
- "Auxiliary Heaters Configuration Tab" on page 89

The configuration window includes the following common buttons:

Get — Click Get to import the current configuration settings. Click OK to save any modifications to the settings.

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

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

**Help** — Use this button to open the help file.

# **General Configuration Tab**

Figure 91. TRACE 1600 Series Configuration

RACE 1600 Series Con	figuration				_		×
Auxiliary Heaters	Oven   Front Inlet   Back	Inlet Front [	Detector Back Detector	Auxiliary Carriers Valves	& Events   S	ignals	1
Device Name: Network Address:	GC 169.254.250.5	— —	Pressure Unit: Cryo: GC Name:	psi	•		
Device Model: Simulation Mode: Simulation File:	TRACE 1600/1610   Off C Read  Trace1600Demo	C Write	Line Frequency: Serial Number: Firmware Version:	60 720009073 00.00.80	Hz		
C Lock the GC front	t panel						
			H2 Sensor Instal	led			
Get				OK	Cancel	Help	

This tab allows you to configure the network address, handshaking parameters, and the pressure units. This tab includes the following fields:

- **Device Name** Set the name of the device to configure.
- Network Address Enter the I.P. address to allow the LAN control of the GC through the Thermo Scientific CDS.

The TRACE 1600 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
- **Timeout (s)** Lists the timeout value in seconds. Generally, the two default values are **500** and **2551** respectively.
- Device Model Select either TRACE 1600/1610 or TRACE 1300/1310.
- Simulation Mode Set to Off, Read, or Write.

- Simulation File Select the simulation file to write to.
- Lock GC Front Panel Check this box to lock the GC front panel.
- Pressure Unit From the drop-down list, choose one: psi, kPa, or bar.
- Cryo Select the coolant used by your cryogenic option. Choose one: None, Liquid Nitrogen or Carbon Dioxide.
- GC Name— Specify a name for the GC in use.
- Line Frequency Select the AC power frequency (50 Hz or 60 Hz) that your GC is plugged into. The line frequency indicates allowable scan rate values.
- Serial Number— Lists the hardware serial number.
- Firmware Version— Lists the firmware version installed on the GC.
- H2 Sensor Installed Select the H2 sensor check box if an H2 sensor is installed in the GC oven.

# **Instrument Configuration Tab**

Autosampler Configuration — If two AI/AS sampling systems are installed on the same GC timebase in the Instrument Configuration Window, use this setting to perform different sample sequences simultaneously on both channels. Select Single, Confirmation, or High Throughput.

- Front Instrument Select the autosampler installed to the front injector of the GC.
- Back Instrument Select the autosampler installed on the back injector of the GC.
- Enable Flexible Dual Control Select this check box if you want to allow for separate autosampler sequences.
- Dual Timeout Select a value between 0 and 86400 seconds.

## **Oven Configuration Tab**

Use this tab to configure the temperature in the GC oven.

- Maximum Temperature Select a value between 0 to 450 °C.
- Minimum Temperature Select a value between 0 to 450 °C.
- Cryogenics Installed Check this box if a cryogenic module is installed.
- Aux Oven Installed Check this box if the TRACE 1600 Auxiliary Oven is installed.

# **Inlet Configuration Tabs**

Configure the front and back inlets using the appropriate tab.

## **Front Inlet Configuration Tab**

This field includes the following options:

- Installed Check this box if a front injector is installed.
- HeSaver-H2 Safer (1600) Installed Check this box if a Helium Saver-H2 Safer (1600) optional kit is installed on the inlet.
- Helium Saver Module (1300) Installed Check this box if a Helium Saver (1300) module is installed on the inlet.
- Name Type a name for the front inlet.
- Type Choose one: None, S/SL, S/SL with Backflush, Gas Sampling Valve, Helium Saver S/SL, PTV, PTV Backflush, or TSI.
- Column Name Add a name for the column.
- Gas Mode— Available when the Helium Saver-H2 Safer optional kit is installed. Choose Standard, HeliumSaver, or Hydrogen Safer.
- **Pressurization Gas**—Available when the Helium Saver-H2 Safer optional kit is installed. Choose **Helium**, **Nitrogen**, or **Argon**.
- Carrier Gas Choose the type of carrier gas to supply the front inlet. Choose one: Helium, Hydrogen, Nitrogen, Argon, or Argon/Methane.
- Backflush type Choose Disable, PreColumn, MidColumn, or PostColumn.
- **Backflush Option Installed** Select this check box if a backflush option is installed on the GC.
- Thermo Spray Injection Installed Select this check box if a Thermo Spray injector is installed on the GC.
- Gas Sampling Valve installed Select this check box if a Gas Sampling Valve injector is installed on the GC.
- Maximum Temperature Select a value between 0 to 450 °C
- Minimum Temperature Select a value between 0 to 450 °C
- Maximum Pressure Select a value between 0.000 to 100.0000 psi.
- Maximum Flow Select a value between 0.001 to 100.00 ml/min.
- Maximum Pressure Rate —Select a value in the allowable range for psi, kPa, or bar.

## **Back Inlet Configuration Tab**

- Installed Check this box if a front injector is installed.
- Helium Saver Module Installed Check this box if a Helium Saver module is installed on the inlet.
- Name Type the name for the back inlet.
- Type Choose one: None, S/SL, S/SL with Backflush, Gas Sampling Valve, Helium Saver S/SL, PTV, and PTV Backflush.
- Column Name Enter a name of the column.
- Carrier Gas Choose the type of carrier gas to supply the front inlet. Choose one: Helium, Hydrogen, Nitrogen, Argon, or Argon/Methane.
- Maximum Temperature Select a value between 0 to 450 °C
- Minimum Temperature Select a value between 0 to 450 °C
- Maximum Pressure Select a value between in allowable range for psi, kPa, or bar.
- Maximum Flow Select a value between 0.001 to 100.00 ml/min.
- Maximum Pressure Rate Select a value between 0.001 to 10.0000 bar/min.
- Backflush type Choose Disable, PreColumn, MidColumn, or PostColumn.
- Backflush Option Installed Select this check box if a backflush option is installed on the GC
- Cryogenics installed Select this check box if a cryogenics module is installed on the GC.

# **Detector Configuration**

Configure the front and back detectors using the appropriate tab.

## **Front Detector Configuration Tab**

Use this tab to configure the detector you will use with your TRACE 1600/1610.

If you change the configuration, remember to reconfigure your TRACE 1600/1610 here.

This tab includes the some of following fields depending on what type of detector is installed:

- Installed Check this box if a front detector is installed.
- Name Enter a name for the front detector.

- Type Choose the type of front detector module (FID, TCD, ECD, NPD, FPD, GDI, PDD, Generic, or None) installed on the GC.
- Maximum Temperature Select a value between 0 to 450 °C
- Minimum Temperature Select a value between 0 to 450 °C
- Maximum Fuel Flow Select a value according to the detector installed.
- Maximum Util Flow Select a value according to the detector installed.
- Maximum Makeup Flow Select a value according to the detector installed.
- Makeup Gas Options depend on the detector installed.
- Signal Press this button to set the Unit, Factor, and Offset settings.

## **Back Detector Configuration Tab**

Use this tab to configure the detector you will use with your TRACE 1600/1610.

If you change the configuration, remember to reconfigure your TRACE 1600/1610 here.

This tab includes the some of following fields depending on what type of detector is installed:

- Installed Check this box if a back detector is installed.
- Name Enter a name for the front detector.
- Type Choose the type of front detector module (FID, TCD, ECD, NPD, FPD, GDI, PDD, or Generic) installed on the GC.
- Maximum Temperature Select a value between 0 to 450 °C
- Minimum Temperature Select a value between 0 to 450 °C
- Maximum Fuel Flow Select a value according to the detector installed.
- Maximum Util Flow Select a value according to the detector installed.
- Maximum Makeup Flow Select a value according to the detector installed.
- Makeup Gas Options depend on the detector installed.
- Signal Press this button to set the Unit, Factor, and Offset settings.

## Aux Left/Rigth Detector Configuration Tab

Use this tab to configure any auxiliary detectors you will use with your TRACE 1600/1610.

If you change the configuration, remember to reconfigure your TRACE 1600/1610 here.

This tab includes the some of following fields depending on what type of detector is installed:

- Installed Check this box if a back detector is installed.
- Name Enter a name for the front detector.
- Type Choose the type of front detector module (FID, TCD, ECD, NPD, FPD, GDI, PDD, or Generic) installed on the GC.
- Maximum Temperature Select a value between 0 to 450 °C
- Minimum Temperature Select a value between 0 to 450 °C
- Maximum Fuel Flow Select a value according to the detector installed.
- Maximum Util Flow Select a value according to the detector installed.
- Maximum Makeup Flow Select a value according to the detector installed.
- Makeup Gas Options depend on the detector installed.
- Signal Press this button to set the Unit, Factor, and Offset settings.

# **Auxiliary Carrier Configuration Tab**

- Installed Check this check box if an Auxiliary Carrier module is installed.
- Enable Enhanced Mode Select this check box if you want to operate in back-pressure mode.
- Auxiliary carrier module 1 Select this check box to enable settings for the auxiliary control module and options installed on your GC or TRACE 1600 Auxiliary Oven.
  - AuxCarrier1 Select the Installed check box and set the location: Front or Back.
     Select the type of the carrier gas in use for the selected auxiliary carrier line 1. The choices are: Helium, Hydrogen, Nitrogen, Argon, or Argon/Methane.
  - AuxCarrier2 Select the Installed check box and set the location: Front or Back.
     Select the type of the carrier gas in use for the selected auxiliary carrier line 2. The choices are: Helium, Hydrogen, Nitrogen, Argon, or Argon/Methane.
  - AuxCarrier3 Select the Installed check box and set the location: Front or Back.
     Select the type of the carrier gas in use for the selected auxiliary carrier line 3. The choices are: Helium, Hydrogen, Nitrogen, Argon, or Argon/Methane.
- Auxiliary carrier module 2 Select this check box to enable settings for the second auxiliary control module and options installed on your GC or TRACE 1600 Auxiliary Oven.
  - AuxCarrier4 Select the Installed check box and set the location: Front or Back.
     Select the type of the carrier gas in use for the selected auxiliary carrier line 4. The choices are: Helium, Hydrogen, Nitrogen, Argon, or Argon/Methane.

- AuxCarrier5 Select the Installed check box and set the location: Front or Back.
   Select the type of the carrier gas in use for the selected auxiliary carrier line 5. The choices are: Helium, Hydrogen, Nitrogen, Argon, or Argon/Methane.
- AuxCarrier6 Select the Installed check box and set the location: Front or Back.
   Select the type of the carrier gas in use for the selected auxiliary carrier line 6. The choices are: Helium, Hydrogen, Nitrogen, Argon, or Argon/Methane.

# **Valves & Events Configuration Tab**

- Event1 to Event8— Select the Installed check box and set the location: Front or Back. Set the Valve Type to Switching or Sampling.
- AuxOvenEvent1 to AuxOvenEvent8— Select the Installed check box and set the location: Front or Back. Set the Valve Type to Switching or Sampling.

# **Signals Configuration Tab**

These controls allow you to configure and use the Signals interface.

- Remote start (IN) Allows another device to start the GC. The choices are High -> Low or Low -> High.
- Inhibit Ready (IN)— Delays readiness until the GC receives a signal from another device. The choices are None, When Low, or When High.
- End of run (OUT) Signals another device that the run has ended. The choices are High -> Low or Low -> High.
- Start of run (OUT) Signals another device, that the run has started. The choices are High -> Low or Low -> High.
- Ready (OUT) Signals another device that the GC is ready. The choices are When Low, or When High.
- **Preparation run** (**OUT**) Signals another device that the GC is preparing for a run. The choices are **When Low**, or **When High**.
- Use default settings Press this button to set the handshaking values back to the original settings.

# **Auxiliary Heaters Configuration Tab**

These controls allow you to configure and use an Auxiliary Heater.

This tab includes the following fields:

- Auxiliary Heaters Enables setting the auxiliary control module and options installed on your GC.
  - Heater 1
    - Name— Name of the heater.
    - Type Select Aux temperature #1 or Transfer line 1.
    - Location Choose Front, Back, or Shared.
  - Heater 2
    - Name— Name of the heater.
    - Type Select Aux temperature #2 or Transfer line 2.
    - Location Choose Front, Back, or Shared.
- Aux Oven Heaters Enables setting the auxiliary control module and options installed on your TRACE 1600 Auxiliary Oven.
  - Heater 1
    - Name— Name of the heater.
    - Type Select Aux temperature #3, AuxOven, or High temperature aux oven.
    - Location Choose Front, Back, or Shared.
  - Heater 2
    - Name— Name of the heater.
    - Type Select Aux temperature #4, Aux column oven, or Methanizer.
    - Location Choose Front, Back, or Shared.

# 5

# **Method Parameter Setup from CDS**

This chapter contains instructions to edit the parameters (according to the front/back inlet, front/back/ detector modules, and optional devices installed), through Chromeleon or SII Thermo Scientific Chromatography Data System (CDS).

#### Contents

- Edit Method Parameters
- GC Oven Settings
- GC Columns
- GC Inlets
- GC Detectors
- GC Aux Heaters
- GC Aux Carrier Settings
- GC Relays and Switching/Sampling Valves
- Retention Time Alignment

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

## **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 1600/1610, you are ready to set the controls to run your method.

# **GC Component Selection/Fluidic Setup**

This page displays components you can configure for the GC, such as inlets, columns, and detectors. Select/clear the check box of a module to determine whether it is fluidically connected to the GC or not.

The following additional parameters appear for the TRACE 1600/1610 :

- Send parameters only (don't run the GC) If selected, the TRACE 1600/1610 will not start when the method runs. The following additional check boxes are enabled:
  - **Specify oven parameters** This option is selected by default. The Oven page will appear in the wizard and oven parameters will be included. If you clear this option, the Oven page will not appear in the wizard and oven parameters will be excluded from the method.
  - Specify relay and valve events This option is selected by default. The Relay/Valve
    and Sampling Valve Event pages will appear in the wizard. If you clear this option,
    the Relay/Valve and Sampling Valve Event pages will not appear in the wizard and
    relay valve and sampling valve events will be excluded from the method.
  - Specify aux heater parameters This option is selected by default. The Aux Heaters
    page will appear in the wizard and parameters will be included. If you clear this
    option, the Aux Heaters page will not appear in the wizard and aux heater parameters
    will be excluded from the method.
- Use default values for all TRACE 1600/1610 modules Select this option to use default values for the TRACE 1600/1610. If you do not select this option, values from the last method run are used.

In the Instrument Method Wizard, only the pages of the selected modules are shown.

# **GC Oven Settings**

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

#### **GC Oven Settings**

The following parameters are editable.

- **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.
- Oven 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 enabled if Oven cryogenics has been selected in Instrument Configuration. See "Valves & Events Configuration Tab" on page 89.
   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 enabled if Oven cryogenics has been selected in Instrument Configuration. See "Valves & Events Configuration Tab" on page 89. Specify the temperature at which the cryogenic system begins to supply the coolant. Enter a value in the range of 40-200 °C.

#### **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 displays a flat line.

• Mode— Select from Off, Constant temperature, or Ramped temperature.

#### 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** 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 Insert Row button automatically adds a ramp level after the last one listed.
  - Clicking on Delete Row 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.
- Target Value— 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.

# **GC Columns**

Select a tab to configure the front or back column.

- Validate Column Configuration— Check this box to verify column configuration.
- Description— Name for the column.
- Length Length of the column.
- Diameter Inner diameter of the column.
- Film thickness— Specifies the film thickness of the column.
- Pre column installed— Select Yes or No.
  - Length Column length.
  - Diameter Inner diameter of the column.
- Post column installed— Select Yes or No.
  - Length Column length.
  - Diameter Inner diameter of the column.

# **Flow and Pressure Options**

Mode— Use this field you select the flow mode for the carrier gas. The choice you select here determines the inlet parameters available Method Editor.

• **Constant Flow** — A single Flow field is available and the graph shows a horizontal line at the flow rate indicated.

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

• **Ramped Flow** — The Flow program fields are displayed. The graph represents the ramps in the program.

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

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.
- Target Value— 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.
- **Constant Pressure** A single Pressure field is available and the graph shows a horizontal line at the pressure indicated.

Use this field to set up pressure when the **Constant pressure** carrier mode has been selected. Enter a value in the range of 0-1050 kPa (0-152 psi; 0-10.50 bar). If you clear the field, the field is disabled and the graph becomes a horizontal line at zero pressure.

• **Ramped Pressure** — The Pressure program fields are displayed. The graph represents the ramps in the program.

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

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.
- Target Value— Enter a value for the constant pressure field of the ramp in the range of 0-1050 kPa (0-152 psi; 0-10.50 bar).
- Hold Time Enter a time for which the pressure will be held. The range is 0.00-999.99 min.
- **Constant Linear Velocity** Constant linear velocity is a carrier control mode where the linear velocity of the carrier gas stays constant through the column. Enter a value in the range of 0.100-1000.000 cm/s.

As the linear velocity depends on the GC Oven temperature, the control will adapt the head pressure of the column during a GC Oven temperature program run in order to keep constant the linear velocity.

# **GC** Inlets

Click the inlet to view the editable parameters:

"S/SL" on page 96

"S/SL Backflush" on page 98

"PTV" on page 100

"PTV Backflush" on page 104

"GSV" on page 109

"Helium Saver (HeS-S/SL)" on page 112

## S/SL

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

## **SSL** Parameters

This page includes the following fields:

#### **Temperature Settings**

• Enable temperature control— Select the check box to enable the adjacent field. Enter a value for the inlet temperature in the range of 0-400 °C.

#### **Inlet Parameters**

Split mode— Select the check box to enable the drop down box.

- 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.
- Split w/Surge Enables all fields. Same as Split but you 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.
- Splitless w/Surge Enables all fields. Same as Splitless but you 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.
- 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 0-1050 kPa (0-152 psi; 0-10.50 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**.

Split flow control— Select the check box to enable the buttons and fields.

Split Flow — Select the check box to enable the adjacent field. Enter a value in the range of 1-1250 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:
  - Mode is set to Split.
  - The Split Ratio check box is selected.

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.

- **Purge flow control** Select the check box to enable these function. 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.
  - Septum 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 For 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.
- Vacuum Compensation Select the check box to enable the vacuum compensation feature. Use this only when the TRACE 1600/1610 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.

• Enable Gas Saver Mode— 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 1-500 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

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

## S/SL Backflush Parameters

#### **Temperature Settings**

• Enable temperature control— Select the check box to enable the adjacent field. Enter a value for the inlet temperature in the range of 0-400 °C.

#### **Inlet Parameters**

Split mode— Select the check box to enable the drop down box.

- 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.
- Split w/Surge Enables all fields. Same as Split but you 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.
- Splitless w/Surge Enables all fields. Same as Splitless but you 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.

- 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 0-1050 kPa (0-152 psi; 0-10.50 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**.

Split flow control— Select the check box to enable the buttons and fields.

Split Flow — Select the check box to enable the adjacent field. Enter a value in the range of 1-1250 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:
  - Mode is set to Split.
  - The Split Ratio check box is selected.

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.

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

- **Purge flow control** Select the check box to enable these function. 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.
  - Septum 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.

	<ul> <li>Stop Purge For — 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.</li> </ul>
	This field includes the following parameters:
	• Vacuum Compensation — Select the check box to enable the vacuum compensation feature. Use this only when the TRACE 1600/1610 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.
	• <b>Gas Saver</b> — 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:
	<ul> <li>Gas Saver Flow — Enter a value for the gas saver flow rate in the range of 1-500 mL/min.</li> </ul>
	<ul> <li>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.</li> </ul>
Backflush	
	Use this field to set up the Backflush parameters.
	• Enable Backflush— Select this check box to enable the Backflush field.
	• <b>Backflush Start Time</b> — Set time at which the backflush starts. Enter a time between 0 and 999.99 min.
	• Enable Custom Duration — Enable control of the inlet On/Off value.
	• Custom Duration— Enter a time between 0 and 999.99 min.
ΡΤν	
	This page is the method editor for the Front/Back Programmable Temperature Vaporizing injector module.

## **PTV** Parameters

#### **Temperature Settings**

• Enable temperature control— Select the check box to enable the adjacent field. If the cryogenic option is present, enter a value for the inlet temperature in a 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.

The Cryogenics parameters are:

- Enable Cryogenics 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.

#### **Inlet Parameters**

• **Split Mode** — 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 Split w/surge Same as Split 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.
- 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.

The following are enabled when the Mode is set to **CT Split w/Surge or CT Splitless** w/Surge.

- Surge Pressure This 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 0-1050 kPa (0-152 psi; 0-10.50 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.
- Split Flow Select the check box to enable the adjacent field. Enter a value in the range of 1-1250 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:
  - Mode is set to PTV Split or CT Split.
  - The Split Ratio check box is selected.

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.

**Purge Flow Control** — 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.

- Septum 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.
- Vacuum Compensation Select the check box to enable the vacuum compensation feature. Use this only when the TRACE 1600/1610 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 1-500 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.

#### **PTV Ramp Settings**

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 the Enable 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 the **Enable clean 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.
- Enable Evaporation Phase Select this check box to enable the Evaporation phase parameters in the Injection Phases field.

- Enable Clean 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, or 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 0-1050 kPa (0-152 psi; 0-10.50 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.

## **Display Phase Program Plot**

To view a graph in the PTV phases dialog box, click **Display phase program plot**. 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.

## **PTV Backflush**

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

## **PTV Backflush Parameters**

#### **Temperature Settings**

• Enable temperature control— Select the check box to enable the adjacent field. If the cryogenic option is present, enter a value for the inlet temperature in a 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.

The Cryogenics parameters are:

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

#### **Inlet Parameters**

• **Split Mode** — 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 Split w/surge Same as Split 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.
- 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.

The following are enabled when the Mode is set to **CT Split w/Surge or CT Splitless** w/Surge.

- Surge Pressure This 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 0-1050 kPa (0-152 psi; 0-10.50 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.
- Split Flow Select the check box to enable the adjacent field. Enter a value in the range of 1-1250 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:
  - Mode is set to PTV Split or CT Split.
  - The Split Ratio check box is selected.

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.

- **Purge Flow Control** 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.
  - Septum 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. This field includes the following parameters:
- Vacuum Compensation Select the check box to enable the vacuum compensation feature. Use this only when the TRACE 1600/1610 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 1-500 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 Enable 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 Enable clean 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.
- Enable Evaporation Phase Select this check box to enable the Evaporation phase parameters in the Injection Phases field.
- Enable Clean Phase Select this check box to enable the Cleaning phase parameters in the Injection Phases field.
- Enable Pressure Ramps— 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 Delay Time— 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 from Turn Off, Cool Down, or 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 0-1050 kPa (0-152 psi; 0-10.50 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.
#### **Display Phase Program Plot**

• To view a graph in the PTV phases dialog box, click **Display phase program plot**. 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.

- Enable Backflush— Select this check box to enable the Backflush field.
- Backflush Start Time— Set time at which the backflush starts. Enter a time between 0 and 999.99 min.
- Enable Custom Duration Enable control of the inlet On/Off value.
- Custom Duration— Enter a time between 0 and 999.99 min.

### GSV

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

## **GSV** Parameters

#### **Temperature Settings**

• Enable 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.

#### **Inlet Parameters**

- Split mode— Select the check box to enable the drop down box.
  - 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 111.

- 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 111.

 Split w/Surge — Enables all fields. Same as Split but you 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 **Split w/Surge** mode is selected the Gas Sampling Valve parameters in the **Valve** field are enabled. See "Valve" on page 111.

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 111.

• **Split Flow** — Select the check box to enable the adjacent field. Enter a value in the range of 1-1250 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:
  - Mode is set to Split.
  - The Split Ratio check box is selected.

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.

This field is enabled when the S/SL Mode is set to Splitless, Split w/ Surge, 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 0-1050 kPa (0-152 psi; 0-10.50 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.

This field includes the following parameters:

- Vacuum Compensation Select the check box to enable the vacuum compensation feature. Use this only when the TRACE 1600/1610 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 1-500 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)

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

# **HeS-S/SL** Parameters

This page includes the following fields:

#### **Temperature Settings**

• Enable temperature control— Select the check box to enable the adjacent field. Enter a value for the inlet temperature in the range of 0-400 °C.

#### **Inlet Parameters**

Split mode— Select from the drop down box.

- 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.
- Split w/Surge Enables all fields. Same as Split but you 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.
- Splitless w/Surge Enables all fields. Same as Splitless but you 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.

- 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 0-1050 kPa (0-152 psi; 0-10.50 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**.

Split flow control— Select the check box to enable the buttons and fields.

Split Flow — Select the check box to enable the adjacent field. Enter a value in the range of 1-1250 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:
  - Mode is set to Split.
  - The Split Ratio check box is selected.

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

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.

- **Purge flow control** Select the check box to enable these function. 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.
  - Septum 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 For — 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.

This field includes the following parameters:

- Vacuum Compensation Select the check box to enable the vacuum compensation feature. Use this only when a TRACE 1600/1610 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 between of 1-500 mL/min.
- Gas Saver Time This is time into the run when the gas saver feature starts up. Enter a value between 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.

# **GC Detectors**

Click the dectector to view the editable parameters:

"FID Page" on page 115

- "ECD Page" on page 116
- "NPD Page" on page 117
- "TCD Page" on page 118
- "FPD Page" on page 119
- "PDD Page" on page 120
- "GDI Page" on page 121
- "Generic" on page 122

## **Detector Time Program**

Select **GC Detectors** for the **Front** or **Back Detector Time Program** tabs, choose the event to be applied. The actions are:

- Time— Enter the time of the event in the detector program.
- Timed Autozero Applies to all detector types. Select this option to zero the detector output at the given time.
- **Timed Polarity** Applies to TCD only. The entry will invert the polarity at the given time. Choose **Postive** or **Negative**.
- Timed Bead Current Applies to NPD only. The entry will change the source current at the given time. Enter a value between 1.0 and 5.0 A.
- Timed Flame Control— Applies to FID and FPD only. Sets time for a flame on/off state. Choose On or Off.
- Timed Pulse Generator—Applies to PDD only. Set times to enable/disable the pulse generator during a run. Choose On or Off.

To create a table, click the buttons on the right:

- Add Appends a line after the last line in the table.
- Insert —Inserts a line above the selected line.
- **Remove** Deletes the selected line.
- Sort —Sorts all lines in the table in ascending order, based on the retention time.

### **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:

#### **Signal Settings**

Data collection rate— Select a value between 1 to 300 Hz.

#### **Detector Settings**

Set the detector parameters in this field.

• Detector temperature control— 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.

	• Flame — 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.
	• Flameout Retry — Select the check box to enable this feature.
	• Enable 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.
	• GC Peak width — Specify the expected width of your GC peaks. For normal chromatography, select <b>Standard</b> . For ultra-fast chromatography select <b>Fast 1</b> . Because this parameter affects the data acquisition rate, selecting Fast for standard chromatography can result in noisier peaks.
Gas Settings	
	These check boxes allow you to turn the flows on and off only.
	• Air flow control— 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.
	• Make-up gas flow control— 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.
	• Hydrogen flow control— 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.
ECD Page	
	ECD Page is the method editor for the Front/Back/Aux L/Aux R Electron Capture Detector module.
Signal Settings	
	Data collection rate— Select a value between 1 to 300 Hz.
Detector Settings	
	Set the detector parameters in this field.
	• Detector temperature control— 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 ECD 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.
	• 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
	n the range 0.1-2.0 μs.

- **Reference Current** Enter a value for the cell reference current in the range of 0.1-1.5 nA.
- GC Peak width Specify the expected width of your GC peaks. For normal chromatography, select **Standard**. For ultra-fast chromatography select **Fast**. Because this parameter affects the data acquisition rate, selecting Fast for standard chromatography can result in noisier peaks.

#### **Gas Settings**

The Makeup check box allows you to turn the flow on and off only.

• Make-up gas flow control— 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.

### **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 Settings**

Set the detector parameters in this field.

- Detector temperature control 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.
- Source Saver Check this box to enable the source saver function intended to reduce the consumption of the NPD source.
- Source Current Value— This is the current applied to heat the thermionic source. Enter a value in the range of 1.000-5.000 A.
- Use Default Polarization Voltage Select the check box to enable this feature. When selected, the Polarization voltage field is not enabled.
- Polarization Voltage Enter a value in the range of 1-100 V.
- GC Peak width Specify the expected width of your GC peaks. For normal chromatography, select **Standard**. For ultra-fast chromatography select **Fast**. Because this parameter affects the data acquisition rate, selecting Fast for standard chromatography can result in noisier peaks.

### **Gas Settings**

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

	• Air flow control— 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 flow control— 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 flow control— 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.
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:
Signal Settings	
	Data collection rate— Select a value between 1 to 300 Hz.
	Polarity— Select Positive or Negative.
Detector Settings	
	Set the detector parameters in this field.
	• Detector temperature control— 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.
	• Enable Filament— 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:
	<ul> <li>from 50 °C to 100 °C if the carrier gas is helium</li> </ul>
	<ul> <li>100 °C if the carrier gas is nitrogen</li> </ul>

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

#### **Gas Settings**

The TCD gas 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.
- Column 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 from Front, Back, or Other.

# **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:

#### **Signal Settings**

**Data collection rate**— Select a value between 1 to 300 Hz.

#### **Detector Settings**

Set the detector parameters in this field.

- Base Temperature Control— 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 Control— 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.

	• Flame — 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 boxe are cleared automatically	
	• <b>Ignition Threshold</b> — The flame will re-ignite if the signal current drops below the value. Set a value in the range 0.0-10.0 nA.	
	• <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.	
	<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>	
Gas Settings		
	These check boxes allow you to turn the flows on and off only.	
	• Air flow control— Select the check box to switch on the air flow and enable the adja flow rate field. Enter a value in the range of 5-500 mL/min.	
	• Hydrogen flow control— 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.	
PDD Page		
	PDD Page is the method editor for the Front/Back/Aux L/Aux R Pulsed Discharge Detector module.	
Signal Settings		
	Data collection rate— Select a value between 1 to 300 Hz.	
Detector Settings		
	Set the detector parameters in this field.	
	• Detector temperature contol— 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.	
	• <b>Pulse generator</b> — Select the check box to turn on the pulse generator. This ignites the plasma discharge, which is done after stabilization and bakeout.	

• GC Peak width — Specify the expected width of your GC peaks. For normal chromatography, select **Standard**. For ultra-fast chromatography select **Fast**. Because this parameter affects the data acquisition rate, selecting Fast for standard chromatography can result in noisier peaks.

# **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 Settings**

Set the detector parameters in this field.

- Detector temperature control— 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.
- GC Peak width Specify the expected width of your GC peaks. For normal chromatography, select **Standard**. For ultra-fast chromatography select **Fast**. Because this parameter affects the data acquisition rate, selecting Fast for standard chromatography can result in noisier peaks.

#### **Gas Settings**

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

- Gas 1 flow control: 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 flow control: 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 flow control: 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 3 encapsulated flow restrictor.

# Generic

#### **Signal Settings**

Data collection rate— Select a value between 1 to 300 Hz.

#### **Detector Settings**

Set the detector parameters in this field.

• GC Peak width — Specify the expected width of your GC peaks. For normal chromatography, select **Standard**. For ultra-fast chromatography select **Fast**. Because this parameter affects the data acquisition rate, selecting Fast for standard chromatography can result in noisier peaks.

# **GC Aux Heaters**

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

This page includes the following fields:

# **Valve Oven Options**

The Valve Oven options 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.

## Valve Oven Time Program

Select **Valve Oven** to activate the associated fields. In the Valve Oven field, set the condition of the desired valve controlled into the TRACE 1600 Auxiliary Oven (Valve Oven).

Determine the parameters for an auxiliary oven event. You can define events for up to eight externally controlled devices.

- Number Select the number of the event in the range 1-8.
- Valve type Depending on the valve type, select Switching or Sampling.
- Setting Applies to switching valves only. Set the event status On or Off.
- Inject for Applies to sampling valves only. Specify the inject duration in [min].

To create a table, click the buttons on the right:

- Add Appends a line after the last line in the table.
- Insert —Inserts a line above the selected line.
- **Remove** Deletes the selected line.
- Sort —Sorts all lines in the table in ascending order, based on the retention time.

# **GC Aux Carrier Settings**

Define carrier gas parameters for 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:

# **Aux Carrier Flow and Pressure Options**

**Mode**— Use this field you select the flow mode for the carrier gas. The choice you select here determines the inlet parameters available Method Editor.

• **Constant Flow** — A single Flow field is available and the graph shows a horizontal line at the flow rate indicated.

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

• **Ramped Flow** — The Flow program fields are displayed. The graph represents the ramps in the program.

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

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.
- Target Value— 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.
- **Constant Pressure** A single Pressure field is available and the graph shows a horizontal line at the pressure indicated.

Use this field to set up pressure when the **Constant pressure** carrier mode has been selected. Enter a value in the range of 0-1050 kPa (0-152 psi; 0-10.50 bar). If you clear the field, the field is disabled and the graph becomes a horizontal line at zero pressure.

• **Ramped Pressure** — The Pressure program fields are displayed. The graph represents the ramps in the program.

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

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.
- Target Value— Enter a value for the constant pressure field of the ramp in the range of 0-1050 kPa (0-152 psi; 0-10.50 bar).
- Hold Time Enter a time for which the pressure will be held. The range is 0.00-999.99 min.
- **Constant Linear Velocity** Constant linear velocity is a carrier control mode where the linear velocity of the carrier gas stays constant through the column. Enter a value in the range of 0.100-1000.000 cm/s.

As the linear velocity depends on the GC Oven temperature, the control will adapt the head pressure of the column during a GC Oven temperature program run in order to keep constant the linear velocity.

# **Aux Carrier Time Program**

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.
- Time Enter the time of the event in the carrier program.

**Note** All timed events before the inject command are handled as prep-run commands. Timed events that follow the inject command are handled as run time events.

- Time Carrier Control Select On or Off to control the carrier flow.
- Timed Pressure Enter a value in the allowed range to change the nominal pressure.
- Timed Flow Enter a value in the allowed range to change the nominal flow.

To create a table, click the buttons on the right:

- Add Appends a line after the last line in the table.
- Insert— Inserts a line above the selected line.
- **Remove** Deletes the selected line.
- Sort —Sorts all lines in the table in ascending order, based on the retention time.

# **GC Relays and Switching/Sampling Valves**

Use this page to set up valve and external events to occur at the Prep-run or during a run. Event states are displayed on the Run-time settings field and are from selections made in the **Valves and Events** configuration tab.

• Time—Enter the time of the event in the event program in [min].

**Note** All entries at time {**Initial time**} are handled as prep-run events. All timed events that follow the prep-run events are handled as run time events.

• State —Select On or Off to set the state of the timed event.

To create a table, click the buttons on the right:

- Add Appends a line after the last line in the table.
- Insert —Inserts a line above the selected line.
- Remove Deletes the selected line.
- Sort —Sorts all lines in the table in ascending order, based on the retention time.

# **Retention Time Alignment**

If you trim a GC column, the retention times for eluting components will change. To avoid changing processing methods, you can use the Retention Time Alignment tool to modify the carrier gas flow rate or pressure in the instrument method so that the components will continue to elute at the same retention time.

Take note of following points concerning the availability of the Retention Time Alignment tool.

- The tool is available only in the Instrument Method Editor. It is not available in the Instrument Method Wizard.
- The tool is available in the Instrument Method Editor only when the following conditions are met:
  - The carrier gas flow rate or pressure is specified and set to a constant value in the instrument method. Gradient flows are not supported.
  - The oven temperature is specified in the instrument method. Gradient temperatures are supported. The initial temperature is used for the alignment calculation.
  - The vacuum compensation is set to either on or off in the instrument method.
- The tool is unavailable if the carrier gas in use by the column in the GC is set to **ArgonMethane**. You can set the carrier gas in the instrument's configuration using Instrument Configuration Manager.

#### **Column Information**

The column information is read-only because the values are read directly from the instrument (the GC).

**Carrier gas**— Displays the carrier gas in use by the column in the GC (read-only). You can set the carrier gas in the instrument's configuration using Instrument Configuration Manager.

**Pressure units**— Displays the pressure unit in use by the GC (read-only). You can set the pressure unit in the instrument's configuration using Instrument Configuration Manager.

**Column length**— Displays the length of the column in the GC (read-only). You can set the column length on the instrument's Inlet ePanel Set.

**Column internal diameter**— Displays the internal diameter of the column in the GC (read-only). You can set the column internal diameter in the instrument's ePanel Set.

Film thickness— Displays the film thickness of the column in the GC (read-only). You can set the column film thickness in the instrument's ePanel Set.

**Precolumn installed**— If a precolumn is installed in the GC, the check box is selected (read-only). If a precolumn is not installed, the check box is not selected. You can switch the Precolumn on or off in the instrument's ePanel Set.

**Precolumn length**— Displays the length of the precolumn if the **Precolumn installed** check box is selected (read-only). You can set the precolumn length in the instrument's ePanel Set.

**Precolumn internal diameter**— Displays the internal diameter of the precolumn if the **Precolumn installed** check box is selected (read-only). You can set the precolumn internal diameter in the instrument's ePanel Set.

#### Method Information

The method information is read-only because the values are read directly from the current instrument method.

**Flow mode**— Displays the flow mode for the carrier gas (read-only), as set in the instrument method. Possible values are **Constant Flow** and **Constant Pressure**.

Flow— (Available only if the flow mode is set to **Constant Flow**.) Displays the flow rate of the carrier gas (read-only), as set in the instrument method.

**Pressure**— (Available only if the flow mode is set to **Constant Pressure**.) Displays the pressure of the carrier gas (read-only), as set in the instrument method.

**Oven temperature**— Displays the oven temperature (read-only), as set in the instrument method.

Vacuum compensation— If vacuum compensation is enabled for the GC in the instrument method, the check box is selected (read-only). If a vacuum compensation is not enabled, the check is not selected.

#### **Actual Column Void Time and Reference Retention Time**

Void time determination method— Select the method to determine the void time.

**Note** The void time is the time taken for the mobile phase and non-retained solutes to pass through the column.

The available options are:

Measured — Select this option if you can measure the void time yourself. In this case, you must enter a value for the void time you have measured in the Measured void field.

Extrapolated — Select this option if you want the void time to be determined by extrapolation using values you enter for Measured retention time (nC8), Measured retention time (nC9), and Measured reference retention time (nC10).

Theoretical void time— Displays the theoretical void time (read-only) calculated using the column and instrument method information (carrier gas type, column dimensions, flow/pressure, and oven temperature).

Measured void time— (Available only if the Void time determination method is set to Measured.) Enter the void time you have measured.

**Extrapolated void time**— (Available only if the **Void time determination method** is set to **Extrapolated**.) Displays the extrapolated void time (read-only). This is determined by extrapolation using the values you enter for Measured retention time (nC8), Measured retention time (nC9), and Measured reference retention time (nC10).

Measured retention time (nC8)— (Available only if the Void time determination method is set to Extrapolated.) Enter the retention time you have measured for pure n-Octane (n-C8).

Measured retention time (nC9)— (Available only if the Void time determination method is set to Extrapolated.) Enter the retention time you have measured for pure n-Nonane (n-C9).

Measured reference retention time (nC10)— Enter the reference retention time you have measured for pure n-Decane (n-C10).

#### **Target Reference Retention Time**

**Target reference retention time (nC10)**— Enter the reference retention time for pure n-Decane (n-C10) in the target data.

#### **Calculated Flow/Pressure**

**Calculated flow/pressure (for method)**— Displays the calculated flow rate or pressure value (read-only) for the carrier gas that must be used in the instrument method in order to align the chromatogram. If the flow mode is set to **Constant Flow**, the calculated flow rate is displayed. If the Flow mode is set to **Constant Pressure**, the calculated pressure is displayed.

**Calculated pressure/flow (information only)**— Displays the corresponding calculated pressure or flow rate value (read-only) for your information only. If the Flow mode is set to Constant Flow, the calculated pressure is displayed. If the Flow mode is set to Constant Pressure, the calculated flow rate is displayed.

#### Align Method

Align Method— Click this button to update the current instrument method with the calculated flow rate or pressure value required to align the chromatogram. If the flow mode is set to Constant Flow, the instrument method is updated with the calculated flow rate. If the Flow mode is set to Constant Pressure, the instrument method is updated with the calculated pressure. The Retention Time Alignment tool closes when you click the button.

# **Oven**

This chapter provides instructions 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.

## 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 Instant Connect 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 92. Split/Splitless Injector Module

The module and injector components are shown in Figure 93 and Figure 94.



Figure 93. 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.





# **Surged Split Mode**

In this mode, a carrier gas pressure surge activates during the injection phase for a preset time (surge duration). The pressure applied (surge pressure) produces a surge of the flow in the injector, and accelerates the transfer process of the sample substances from the injector to the column.

# **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.



**Figure 96.** Splitless Mode Block Diagram

# **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 1610 autosampler.

# **LV Splitless Injection Requirements**

Large Volume-Splitless injection requires the SSL-LV Kit for TRACE 1600/1610 (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 re-condensation is shown in Figure 97.

Figure 97. 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 1600/1610. 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<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 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	A	Deactivated Single Tapered empty liner.

#### **HS/SPME** Liners

Select an appropriate liner from Table 7.

Table 7.	<b>HS/SPME</b> Liners
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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 1600/1610 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 split, and surged splitless; and the flow mode: constant flow, constant pressure, ramped flow, ramped pressure, and constant linear velocity.

- Carrier Gas Parameters
- Injection Mode
- Inlet Parameters
- Purge Parameters
- Surge Parameters
- Setting Gas Parameters
- Setting Parameters for the Split Mode
- Setting Parameters for the Surged 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; 0-1050 kPa (0-152 psi; 0-10.50 bar). This line is not editable when Ramped Flow or Ramped 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 Constant Pressure, Ramped Pressure, or Constant Linear Velocity 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.
- **Ramped 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.
- **Ramped 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.
- **Constant Linear Velocity** Constant linear velocity is a carrier control mode where the linear velocity of the carrier gas stays constant through the column. Enter a value in the range of 0.100-1000.000 cm/s.

As the linear velocity depends on the GC Oven temperature, the control will adapt the head pressure of the column during a GC Oven temperature program run in order to keep constant the linear velocity.

**Linear Velocity** — The calculated velocity of the carrier gas through the column, expressed in cm/s. It is editable when **Constant Linear Velocity** is selected.

**Void Time** — The elution time of an un-retained peak, expressed in seconds. It is not editable.

**Gas Saver Flow** — This function reduces carrier gas consumption. The range is On/Off; 1–500 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 1600/1610 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.

**Surged Split**— Same as **Split** but can also program a surge during an injection. Surge starts during the injection phase and continues until the surge duration time is finished. Surge is further defined in next group of controls.

**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 1-1250 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 Ramped 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.

**Note** When using the split ratio setting, you must configure the column dimensions or calibrate the column to obtain an accurate split flow.

**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 Split** or **Surged Splitless**.

**Surge Pressure** — Defines the pressure applied during a **Surged Split** or **Surged Splitless** injection. The pressure produces a surge of flow in the injector to speed the transfer of the sample. Enter a value in the range of 0-1050 kPa (0-152 psi; 0-10.50 bar).

**Surge Duration** — Defines the time that the surge pressure is maintained during a **Surged Split** or **Surged Splitless** injection. Enter a value in the range of 0.00-999.99 min. Typically, set to coincide with the **Splitless time** during a **Surged Splitless** injection.

## **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, Constant Linear Velocity, Ramped Flow or Ramped Pressure.
#### c. Enter the initial Flow, Pressure, or Constant Linear Velocity.

- 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.
- iii. If you select Constant Linear Velocity, enter the desired value.
- d. Enter a Ramped Flow/Ramped Pressure.

**Note** When you select Ramped Flow or Ramped Pressure mode, the menu for the carrier contains parameters for up to three program ramps.

- i. Select **Ramped 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 1600/1610 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 (Ramped 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 Surged 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 Surged Split injection

- 1. Program the carrier gas flow.
- 2. From the mode list, choose Surged Split.
- 3. Set the injector **Temperature**.
- 4. If carrier **Flow** mode (Ramped 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 values for Surge Pressure and Surge Duration.
- 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 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.

8

## **Split/Splitless Backflush Injector (SSLBKF) Module**

This chapter describes the Instant Connect 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.

**Note** The metal backflush line ships with a pre-installed metal ferrule and nut.



Figure 98. Split/Splitless Backflush Injector Module

The module and injector components are shown in Figure 99 and Figure 100.



Figure 99. 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 101. 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.

## **Surged Split Mode**

In this mode, a carrier gas pressure surge activates during the injection phase for a preset time (surge duration). The pressure applied (surge pressure) produces a surge of the flow in the injector, and accelerates the transfer process of the sample substances from the injector to the 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 1600/1610. 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<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 8.

Table 8.	Split Liners	
----------	--------------	--

No	Liner	Description
1		Deactivated liner; 4-mm ID; Glass Wool; 900 $\mu$ L 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	7	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 1600/1610 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 split, and surged splitless; and the flow mode: constant flow, constant pressure, ramped flow, ramped pressure, and constant linear velocity.

- 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 Surged 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; 0-1050 kPa (0-152 psi; 0-10.50 bar). This line is not editable when the Constant Flow or Ramped 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 Ramped 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.
- **Ramped 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.
- **Ramped 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.

• **Constant Linear Velocity**— Constant linear velocity is a carrier control mode where the linear velocity of the carrier gas stays constant through the column. Enter a value in the range of 0.100-1000.000 cm/s.

As the linear velocity depends on the GC Oven temperature, the control will adapt the head pressure of the column during a GC Oven temperature program run in order to keep constant the linear velocity.

**Linear Velocity** — The calculated velocity of the carrier gas through the column, expressed in cm/s. It is editable when **Constant Linear Velocity** is selected.

**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; 1–500 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 1600/1610 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.

**Surged Split**— Same as **Split** but can also program a surge during an injection. Surge starts during the injection phase and continues until the surge duration time is finished. Surge is further defined in next group of controls.

**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 1-1250 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 Ramped 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.

**Note** When using the split ratio setting, you must configure the column dimensions or calibrate the column to obtain an accurate split flow.

**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 Split** or **Surged Splitless**.

**Surge Pressure** — Defines the pressure applied during a **Surged Split** or **Surged Splitless** injection. The pressure produces a surge of flow in the injector to speed the transfer of the sample. Enter a value in the range of 0-1050 kPa (0-152 psi; 0-10.50 bar).

**Surge Duration** — Defines the time that the surge pressure is maintained during a **Surged Split** or **Surged Splitless** injection. Enter a value in the range of 0.00-999.99 min. Typically, set to coincide with the **Splitless time** during a **Surged Splitless** injection.

## **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, Constant Linear Velocity, Ramped Flow or Ramped Pressure.
  - c. Enter the initial Flow, Pressure, or Constant Linear Velocity.
    - 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.
    - iii. If you select Constant Linear Velocity, enter the desired value.
  - d. Enter a Ramped Flow/Ramped Pressure.

**Note** When you select Ramped Flow or Ramped 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 1600/1610 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.

```
Note Select the backflush type (pre-, middle, post-column backflush) on the inlet configuration page.
```

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 (Ramped 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 Surged 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 Surged Split.
- 3. Set the injector Temperature.
- 4. If carrier **Flow** mode (Ramped 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 values for Surge Pressure and Surge Duration.
- 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 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 1600/1610 Hardware Manual*.

## 9

## **Gas Sampling Valve (GSV) Module**

This chapter describes the Instant Connect 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 102. Gas Sampling Valve Module



The components of the module are shown in Figure 103.



Figure 103. Gas Sampling Valve Module Components

**IMPORTANT** The valve ships with relief pins. These pins MUST BE removed from the valve body before working with the valve.





Once the actuation tubing has been set up, set the carrier gas input pressure in a range from 450 kPa (65 psig; 4.5 bar) to 690 kPa (100 psi, 6.9 bar), actuate the valve and remove the pins. You may need to increase 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** The carrier gas input pressure must be set in a range of **450 kPa** (**65 psig**; **4.5 bar**) to **690 kPa** (**100 psi**, **6.9 bar**). OTHERWISE, THE VALVE MAY BE DAMAGED.



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 104 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.

## **Packed Column Adapters**

The GSV module is compatible with a packed column adapter (P/N 19050762). To install a packed column, replace the pre-installed capillary column nut with the packed column adapter.

## **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 split, and surged splitless; and the flow mode: constant flow, constant pressure, ramped flow, ramped pressure, and constant linear velocity.

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 Surged 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; 0-1050 kPa (0-152 psi; 0-10.50 bar). This line is not editable when the Constant Flow or Ramped 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 Ramped 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.
- **Ramped 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.
- **Ramped 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.

• **Constant Linear Velocity**— Constant linear velocity is a carrier control mode where the linear velocity of the carrier gas stays constant through the column. Enter a value in the range of 0.100-1000.000 cm/s.

As the linear velocity depends on the GC Oven temperature, the control will adapt the head pressure of the column during a GC Oven temperature program run in order to keep constant the linear velocity.

**Linear Velocity** — The calculated velocity of the carrier gas through the column, expressed in cm/s. It is editable when **Constant Linear Velocity** is selected.

**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; 1–500 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 1600/1610 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 Split** — Same as **Split** 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 enabled.

**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 1-1250 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 Ramped 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.

**Note** When using the split ratio setting, you must configure the column dimensions or calibrate the column to obtain an accurate split flow.

**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 Split** or **Surged Splitless**.

**Surge Pressure** — Defines the pressure applied during a **Surged Split** or **Surged Splitless** injection. The pressure produces a surge of flow in the injector to speed the transfer of the sample. Enter a value in the range of 0-1050 kPa (0-152 psi; 0-10.50 bar).

**Surge Duration** — Defines the time that the surge pressure is maintained during a **Surged Split** or **Surged Splitless** injection. Enter a value in the range of 0.00-999.99 min. Typically, set to coincide with the **Splitless time** during a **Surged Splitless** injection.

## **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**, **Constant Linear Velocity**, **Ramped Flow** or **Ramped Pressure**.
  - c. Enter the initial Flow, Pressure, or Constant Linear Velocity.
    - 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.
    - iii. If you select Constant Linear Velocity, enter the desired value.
  - d. Enter a Ramped Flow/Ramped Pressure.

**Note** When you select Ramped Flow or Ramped 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 1600/1610 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 (Ramped 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 Surged 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 **Surged Split**.
- 3. Set the injector Temperature.
- 4. If carrier **Flow** mode (Ramped 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 values for Surge Pressure and Surge Duration.
- 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 1600/1610 Hardware Manual*.

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# 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 105. Instant Connect Helium Saver Injector Module



The module and injector components are shown in Figure 106 and Figure 107.

## Figure 106. Instant Connect Helium Saver Injector Module Components







Figure 107. Instant Connect Helium Saver Injector Module: Injector Components



**READ MANUAL** Refer to the *TRACE 1600/1610 Hardware Manual* and the *Instant Connect Helium 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 108 on page 178 illustrates the injector module in the default mode of operation.





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 109.

Figure 109. 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 110) 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.





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).

<sup>2</sup> 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 Injection**

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.

- 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 1600/1610. 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		Deactivated Single Tapered empty liner.

**Note** A glass wool liner is recommended for better injection performance and repeatability.

### **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 1600/1610 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 split, and surged splitless; and the flow mode: constant flow, constant pressure, ramped flow, ramped pressure, and constant linear velocity.

- Carrier Gas Parameters
- Injection Mode
- Inlet Parameters
- Purge Parameters
- Surge Parameters
- Setting Gas Parameters
- Setting Parameters for the Split Mode
- Setting Parameters for the Surged 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; 0-1050 kPa (0-152 psi; 0-10.50 bar). This line is not editable when the Constant Flow or Ramped 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 Ramped 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.
- **Ramped 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.
- **Ramped 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.
- **Constant Linear Velocity** Constant linear velocity is a carrier control mode where the linear velocity of the carrier gas stays constant through the column. Enter a value in the range of 0.100-1000.000 cm/s.

As the linear velocity depends on the GC Oven temperature, the control will adapt the head pressure of the column during a GC Oven temperature program run in order to keep constant the linear velocity.

**Linear Velocity** — The calculated velocity of the carrier gas through the column, expressed in cm/s. It is editable when **Constant Linear Velocity** is selected.

**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; 1–500 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 1600/1610 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.

**Surged Split**— Same as **Split** but can also program a surge during an injection. Surge starts during the injection phase and continues until the surge duration time is finished. Surge is further defined in next group of controls.

**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 1-1250 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 Ramped 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.

**Note** When using the split ratio setting, you must configure the column dimensions or calibrate the column to obtain an accurate split flow.

**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 Split** or **Surged Splitless**.

**Surge Pressure** — Defines the pressure applied during a **Surged Split** or **Surged Splitless** injection. The pressure produces a surge of flow in the injector to speed the transfer of the sample. Enter a value in the range of 0-1050 kPa (0-152 psi; 0-10.50 bar).

**Surge Duration** — Defines the time that the surge pressure is maintained during a **Surged Split** or **Surged Splitless** injection. Enter a value in the range of 0.00-999.99 min. Typically, set to coincide with the **Splitless time** during a **Surged Splitless** injection.

### **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**, **Constant Linear Velocity**, **Ramped Flow** or **Ramped Pressure**.
  - c. Enter the initial Flow, Pressure, or Constant Linear Velocity.
    - 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.
    - iii. If you select **Constant Linear Velocity**, enter the desired value.
  - d. Enter a Ramped Flow/Ramped Pressure.

**Note** When you select Ramped Flow or Ramped 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 1600/1610 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 (Ramped 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 Surged 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 Surged Split.
- 3. Set the injector Temperature.
- 4. If carrier **Flow** mode (Ramped 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 values for Surge Pressure and Surge Duration.
- 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 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.

# **Instant Connect Helium Saver-H2 Safer Option**

This guide provides technical information and installation instructions for the Thermo Scientific<sup>m</sup> Instant Connect Helium Saver-H<sub>2</sub> Safer option.

#### Contents

- Overview
- Principle of Operation
- Gas Requirements
- Consumables
- Using Helium Saver-H2 Safer Parameters

# **Overview**

The Instant Connect Helium Saver- $H_2$  Safer option is an innovative Split/Splitless (SSL) inlet system capable of dramatically reducing the consumption of increasingly expensive and difficult-to-procure helium gas. In  $H_2$  Safer mode, the low flow of hydrogen as a carrier gas through the transfer tube allows for safe operations when hydrogen is selected as the carrier 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 1. Instant Connect Helium Saver-H<sub>2</sub> Safer Option

The helium transfer tube acts as the hydrogen transfer tube in the  $H_2$  Safer mode of operation and is connected to a gas net mating block on the top of the GC.



**Figure 2.** Gas Net Mating Block

Figure 3. Rear Connections for Helium Saver-H<sub>2</sub> Safer



The option and injector components are shown in Figure 4 and Figure 5.











#### Figure 6. Helium Saver-H<sub>2</sub> Safer Injector Components

# **Principle of Operation**

In a conventional Split/Splitless injector (SSL), the amount of carrier gas used for chromatographic separation into the analytical capillary column (0.25-0.32 mm ID) is typically 1-2 mL/min, 3-5 mL/min are used for the septum purge flow and most of the carrier gas is consumed through the split line with flow rates up to hundreds of mL/min. Therefore, a standard SSL operation requires the inlet to be supplied with 50-100 mL/min of carrier gas of which only 1-2 mL/min are used to feed the column for the chromatographic separation See Figure 7.



**Figure 7.** Standard SSL Injector Flows

**Note** Running the module in  $H_2$  Safer mode is the same working principle, but with hydrogen flowing instead of helium.

The Helium Saver- $H_2$  Safer works by decoupling the gas used for the chromatographic separation from the gas used to pressurize the inlet and maintain split and purge flows. This allows you to reduce the carrier gas (helium or hydrogen) total flow to the limited value required to carry on the separation process into the analytical column.

Two operative gas modes available are:

- The Helium Saver mode ensures a significant reduction in helium consumption during operation.
- The H<sub>2</sub> Safer mode ensures the safe use of hydrogen as a carrier gas without the need to install a hydrogen sensor in the GC oven.

The core of the Helium Saver-H<sub>2</sub> Safer technology is the modified SSL body supplied by two gas lines:

- The **pressurizing gas line** at the top of the inlet (as the carrier gas line in a standard SSL) provides the required inlet pressure, the septum purge, and split flows. A low-cost, inert gas, like nitrogen, is recommended as a pressurization gas.
- The carrier gas line at the base of the injector body delivers the carrier gas to the column, helium or hydrogen, depending on the configuration and the gas mode selected. A short segment of tubing acts as a back diffusion barrier preventing the pressurizing gas from entering the column during the chromatographic run. See Figure 8.



Figure 8. Helium Saver-H<sub>2</sub> Safer Insert



The carrier gas supply is controlled by a dedicated valve allowing you to set a flow level: 0.1 mL/min and 9 mL/min. This creates two inlet conditions:

• Inlet condition 1—Carrier gas flow set at 0.1 mL/min: the analytical column is filled with the pressurizing gas from the top of the inlet. This minimum carrier gas flow prevents the pressurizing gas from entering the carrier line



Inlet Conditions with the Helium Carrier Gas Flow Set at 0.1 mL/min Figure 9.

• Inlet condition 2—Carrier gas flow set at 9 mL/min: the analytical column is filled with the carrier gas. The column flow rate is controlled by the inlet pressure while the carrier gas flow exceeding the column flow value is diverted upward preventing the back diffusion of the pressurizing gas from the inlet into the column



Figure 10. Inlet Conditions with the Hydrogen Carrier Gas Flow Set at 9 mL/min

The above two states of the injector show the injection phase and the separation processes, as described below:

- Injection phase—The sample is injected into the liner where it vaporizes before being transferred by the gas flow from the inlet chamber into the column. This step is realized with the inlet condition 1: the pressurizing gas sweeps the vaporized analytes and transfer them into the column. See Figure 9. The minimum flow of carrier gas (0.1 mL/min) keeps the gas line free from the injected solvents preventing contamination and possible carryover. According to the injection mode, split or splitless, during this step the sample is totally transferred into the column, or it is split between the column and the split line.
- Separation process—Once the analytes transfer is completed (at the end of the splitless time in splitless mode or after few seconds in split mode) the inlet rapidly switches to condition 2: the carrier gas is supplied at higher flow filling the analytical column and providing the back diffusion barrier that stops the pressurizing gas from entering the column. See Figure 10.

Therefore, the chromatographic separation of the analytes is carried out by the carrier gas at the set flow rate controlled by the inlet pressure, ensuring analytical performances equivalent to a standard SSL inlet but with drastically limited consumption of carrier gas.

# **Helium Saver Mode**

Select **Helium Saver** mode when helium is used as a carrier gas to ensure a significant reduction in helium consumption not only when the GC is idle, but also across all the GC operation phases:

- During the injection phase the helium flow is 0.1 mL/min, regardless the injection mode (split or splitless), being the split flow provided by the pressurizing gas.
- During the analytical run the helium consumption is limited to the 9 mL/min required to feed the column and assure the back-diffusion barrier

The operating principle of Helium Saver technology minimizes helium consumption and is an effective solution to a helium conservation challenge.

### **Difference with the SSL Gas Saver Mode**

The way the **Helium Saver** mode works to reduce helium consumption is substantially different than the **Gas Saver** mode available within standard SSL control parameters. The **Gas Saver** mode is a well-established approach to reduce helium consumption through the split line and it reduces the split flow (typically to 10-15 mL/min) at a specified time after the sample transfer into column, turning it to higher values before the next injection. The resulting gas savings is therefore less effective.

The **Gas Saver** mode is compatible with the Helium Saver- $H_2$  Safer and it can be used to reduce the consumption of pressurizing gas.

# H<sub>2</sub> Safer Mode

The  $H_2$  Safer mode allows for the safe use of hydrogen as a carrier gas without the need to install a hydrogen sensor in the GC oven.

When a standard SSL is used with hydrogen as carrier gas, it is typically configured to allow hundreds of mL/min of hydrogen flow. This allows the ability to set high split flows and subsequently high split ratios when using split injections. If there is column breakage or a leaking inlet ferrule this causes the oven to rapidly fill with explosive gas; the hydrogen sensor is a mandatory safety requirement to use hydrogen as carrier gas with a standard SSL.

With the  $H_2$  Safer mode, the maximum flow of hydrogen supplied to the inlet is 9 mL/min. In addition, since the inlet is supplied with a much higher flow of inert pressurizing gas, in case of leaks the pressurizing gas dilutes the hydrogen and helps purge it from the oven. Combining the limited hydrogen flow and the dilution of inert gas, an explosive concentration of hydrogen in the GC oven chamber cannot be reached.

In addition, high split ratios are available since the split is accomplished using the pressurization gas.

# **Gas Requirements**

Nitrogen 99.999% with a gas regulator output capable of 100 psig (690 kPa; 6.90 bar).

**Note** It is recommend to use a high capacity oxygen trap with a specification of 15 ppb or less of oxygen in the output.

Helium 99.999% with a gas regulator output capable of 100 psig (690 kPa; 6.90 bar) for Helium Saver mode.

Hydrogen 99.999% with a gas regulator output capable of 60 psig (414 kPa; 4.14 bar) for  $\rm H_2$  Safer mode.

### **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 produce hydrogen in the quantity used in the GC.

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. The lower flammable limit of hydrogen in air is 4%.

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

**IMPORTANT** Use a hydrogen generator that uses a palladium dryer or thermally cycled molecular sieve traps in order to reduce water to trace levels. Silica gel-based dryers are unsuitable for GC carrier gas.

• 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.

# 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 1600/1610. 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.

#### 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 use the **Vapor Volume Calculator** to rapidly calculate the expansion volume based on several factors (solvent, injected liquid volume, temperature, and inlet pressure) to help you determine if a liner dimension is suitable for a method.

**Note** Glass wool-packed liners are recommended for use with the Helium Saver- $H_2$  Safer to reduce possible contamination of the body and cleaning requirements.

#### **Split Liners**

Select an appropriate liner from Table 1.

 Table 1.
 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 2.

Table 2.	Splitless	Liners
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No	Liner	Description
1		Deactivated Single Tapered Glass Wool; 900 µL theoretical volume.
2	A	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. Refer to the *TRACE 1600/1610 Hardware Manual* for information about replacing the O-rings.

# **Using Helium Saver-H2 Safer Parameters**

This menu includes the operating parameters for the Helium Saver- $H_2$  Safer. Editable parameters will vary with the operating mode: split, splitless, surged split, surged splitless, and the flow mode: constant flow, constant pressure, ramped flow, ramped pressure, or constant linear velocity.

- "Carrier Gas Parameters" on page 206
- "Injection Mode" on page 208
- "Inlet Parameters" on page 208
- "Purge Parameters" on page 209
- "Setting Gas Parameters" on page 210
- "Setting Parameters for the Split Mode" on page 211
- "Setting Parameters for the Splitless Mode" on page 212
- "Setting Parameters for the Surged Splitless Mode" on page 212

The following sections list and describe the parameters controlling the front/back SSL injector. They are:

#### Mode

Configure the mode to He Saver, H2 Safer, or Standard.

#### **Carrier Gas Parameters**

Set the carrier gas control parameters. Available parameters change according to the **Flow Mode** set.

**Note** Not all settable flows or pressures are usable for the Helium Saver -  $H_2$  Safer. Refer to the *Helium Saver-H*<sub>2</sub> Safer User Guide for more information about flow rates.

**Pressure** — Defines the actual and setpoint pressure of the carrier gas. The range is On/Off; 0-1050 kPa (0-152 psi; 0-10.50 bar). This line is not editable when the Constant Flow or Ramped 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 Ramped 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.
- **Ramped 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.
- **Ramped 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.
- **Constant Linear Velocity** Constant linear velocity is a carrier control mode where the linear velocity of the carrier gas stays constant through the column. Enter a value in the range of 0.100-1000.000 cm/s.

As the linear velocity depends on the GC Oven temperature, the control will adapt the head pressure of the column during a GC Oven temperature program run in order to keep constant the linear velocity.

**Linear Velocity** — The calculated velocity of the carrier gas through the column, expressed in cm/s. It is editable when **Constant Linear Velocity** is selected.

**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; 1–500 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 1600/1610 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.

**Surged Split**— Same as **Split** but can also program a surge during an injection. Surge starts during the injection phase and continues until the surge duration time is finished. Surge is further defined in next group of controls.

**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 1-1250 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. If the Split Flow value is 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 Ramped 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.

**Note** When using the split ratio setting, you must configure the column dimensions or calibrate the column to obtain an accurate split flow.

**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 a range of 0.5-50 mL/min, must be set in the injector Configuration Page.

**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 Split** or **Surged Splitless**.

**Surge Pressure** — Defines the pressure applied during a **Surged Split** or **Surged Splitless** injection. The pressure produces a surge of flow in the injector to speed the transfer of the sample. Enter a value in the range of 0-1050 kPa (0-152 psi; 0-10.50 bar).

**Surge Duration** — Defines the time that the surge pressure is maintained during a **Surged Split** or **Surged Splitless** injection. Enter a value in the range of 0.00-999.99 min. Typically, set to coincide with the **Splitless time** during a **Surged Splitless** injection.

### **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**, **Constant Linear Velocity**, **Ramped Flow** or **Ramped Pressure**.
  - c. Enter the initial Flow, Pressure, or Constant Linear Velocity.
    - 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.
    - iii. If you select **Constant Linear Velocity**, enter the desired value.
  - d. Enter a Ramped Flow/Ramped Pressure.

**Note** When you select **Ramped Flow** or **Ramped Pressure** mode, the menu for the carrier contains parameters for up to three program ramps.

- i. Select **Ramped 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 1600/1610 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 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 (Ramped 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 **valve delay** is enabled, there is a delay from start of run when the valve in the valve box is switched on.
- 6. If desired, turn on Gas Saver and set Gas Saver Time after the injection time.

#### Setting Parameters for the Surged 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 Surged Split injection

- 1. Program the carrier gas flow.
- 2. From the mode list, choose **Surged Split**.
- 3. Set the injector **Temperature**.
- 4. If carrier **Flow** mode (Ramped 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 values for Surge Pressure and Surge Duration.
- 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 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.

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# **Programmable Temperature Vaporizing Injector** (**PTV**) **Module**

This chapter describes the Instant Connect 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 11. Programmable Temperature Vaporizing Injector Module

The module and injector components are shown in Figure 12 and Figure 13.



Figure 12. Programmable Temperature Vaporizing Injector Modules Components

Figure 13. Programmable Temperature Vaporizing Injector Components



The injector works at constant or programmed temperature 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 14 and Figure 15 respectively.



Figure 14. PTV Cryo System with Liquid Nitrogen

Figure 15. 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, CT Surged Split, 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 16. PTV Split Mode Block Diagram

Figure 17 shows an example of a temperature profile and timing of the valves in Split mode.


Figure 17. PTV Split Mode: Example of Temperature Profile and Timing

# 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 18. PTV Splitless Mode Block Diagram

Figure 19 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.

 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 μL. The initial analytical conditions must be maintained throughout the injections.

Figure 20 shows an example of a temperature profile and timing of the valves in Large Volume mode.





## **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 21 shows an example of a temperature profile and valves timing.

Figure 21. 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 22 shows an example of a temperature profile and valves timing.



Figure 22. CT Splitless Mode Valves Timing

## **Constant Temperature Surged 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.

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.

## **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 223. 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 1610 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 1600/TRACE 1610. 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 3.

 Table 3.
 Split 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		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. 2 mm 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 4.

#### Table 4. 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.

#### Table 4. Splitless Liners

No	Liner	Description
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 5.

#### **Table 5.**Large Volume Liners

No	Liner	Description
1	うた うち うち ふち ふた しち	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 6.

#### Table 6. Like On-column Liner

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 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 the CT Surged 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; 0-1050 kPa (0-152 psi; 0-10.50 bar). This line is not editable when the Constant Flow or Ramped 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 Ramped 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.
- **Ramped 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.
- **Ramped 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.

• **Constant Linear Velocity**— Constant linear velocity is a carrier control mode where the linear velocity of the carrier gas stays constant through the column. Enter a value in the range of 0.100-1000.000 cm/s.

As the linear velocity depends on the GC Oven temperature, the control will adapt the head pressure of the column during a GC Oven temperature program run in order to keep constant the linear velocity.

**Linear Velocity** — The calculated velocity of the carrier gas through the column, expressed in cm/s. It is editable when **Constant Linear Velocity** is selected.

**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; 1–500 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 1600/TRACE 1610 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 Split with Surge**— Same as **Split** 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.

**CT Splitless with 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.

**OnColumn** — 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 1 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 Ramped 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.

**Note** When using the split ratio setting, you must configure the column dimensions or calibrate the column to obtain an accurate split flow.

**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 **CT Split with Surge** or **CT Splitless with Surge**.

**Surge Pressure** — Defines the pressure applied during a **CT Split with Surge** or **CT Splitless with Surge** injection. The pressure produces a surge of flow in the injector to speed the transfer of the sample. Enter a value in the range of 0-1050 kPa (0-152 psi; 0-10.50 bar).

**Surge Duration** — Defines the time that the surge pressure is maintained during a **CT Split** with Surge or **CT Splitless with Surge** injection. Enter a value in the range of 0.00-999.99 min. Typically, set to coincide with the Splitless time during a **CT Splitless with Surge** injection.

#### **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; 0-1050 kPa (0-152 psi; 0-10.50 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 1-1250 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; 0-1050 kPa (0-152 psi; 0-10.50 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; 0-1050 kPa (0-152 psi; 0-10.50 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**, **Constant Linear Velocity**, **Ramped Flow** or **Ramped Pressure**.
  - c. Enter the initial Flow, Pressure, or Constant Linear Velocity.
    - 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.
    - iii. If you select **Constant Linear Velocity**, enter the desired value.
  - d. Enter a Ramped Flow/Ramped Pressure.

**Note** When you select Ramped Flow or Ramped 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 1600/1610 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 233 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 233 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 233 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 23 shows a generic temperature program of the PTV injection cycle.



- **Figure 23.** 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 ramped pressure or ramped 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 the CT Surged 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 CT Surged 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. 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.

## **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.

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# Programmable Temperature Vaporizing Backflush Injector (PTVBKF) Module

This chapter describes the Instant Connect 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.

**Note** The metal backflush line ships with a pre-installed metal ferrule and nut.



Figure 24. PTV Backflush Injector Module

The module and injector components are shown in Figure 25 and Figure 26.



Figure 25. PTV Backflush Module Components



Figure 26. PTV Backflush Injector Components

The injector works at constant or programmed temperatures from ambient temperature up to 450 °C. Up to three temperature rates and plateaus are available when the 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 when 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 dioxide are shown in Figure 27 and Figure 28 respectively.



Figure 27. PTV Cryo System with Liquid Nitrogen

Figure 28. PTV Cryo System with Carbon Dioxide



# **Backflush Mode**

This operating mode eliminates the solvent before the sample enters the column and also, during the cleaning phase, the heavy part of the sample, which is not need for 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 29. PTV Backflush Mode Block Diagram

Figure 30 shows an example of a temperature profile and timing of the valves in Backflush mode.



Figure 30. 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 12, "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 1600/1610. 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 7.

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 8.

Table 8.	Large	Volume	Liners	(Sheet 1	of 2)
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No	Liner	Description
1	タボ うち うた うた みた うた	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 8.** 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 Parameters for Backflush
- Programming Injection Parameters

## **Carrier Gas Parameters**

Set the carrier gas parameters, regardless of the carrier gas. Available parameters change according to the set **Flow Mode**.

**Pressure** — Defines the actual and setpoint pressure of the carrier gas. The range is On/Off; 0-1050 kPa (0-152 psi; 0-10.50 bar). This line is not editable when the Constant Flow or Ramped 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 Ramped 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.
- **Ramped 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.
- **Ramped 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.

• **Constant Linear Velocity**— Constant linear velocity is a carrier control mode where the linear velocity of the carrier gas stays constant through the column. Enter a value in the range of 0.100-1000.000 cm/s.

As the linear velocity depends on the GC Oven temperature, the control will adapt the head pressure of the column during a GC Oven temperature program run in order to keep constant the linear velocity.

**Linear Velocity** — The calculated velocity of the carrier gas through the column, expressed in cm/s. It is editable when **Constant Linear Velocity** is selected.

**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; 1–500 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 1600/1610 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 Split** — Same as **Split** 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.

**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 1 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 Ramped 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.

**Note** When using the split ratio setting, you must configure the column dimensions or calibrate the column to obtain an accurate split flow.

**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 **CT Split with Surge** or **CT Splitless with Surge**.

**Surge Pressure** — Defines the pressure applied during a **CT Split with Surge** or **CT Splitless with Surge** injection. The pressure produces a surge of flow in the injector to speed the transfer of the sample. Enter a value in the range of 0-1050 kPa (0-152 psi; 0-10.50 bar).

**Surge Duration** — Defines the time that the surge pressure is maintained during a **CT Split** with **Surge** or **CT Splitless with Surge** injection. Enter a value in the range of 0.00-999.99 min. Typically, set to coincide with the **Splitless time** during a **CT Splitless with Surge** injection.

#### **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; 0-1050 kPa (0-152 psi; 0-10.50 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; 0-1050 kPa (0-152 psi; 0-10.50 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; 0-1050 kPa (0-152 psi; 0-10.50 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**, **Constant Linear Velocity**, **Ramped Flow** or **Ramped Pressure**.
  - c. Enter the initial Flow, Pressure, or Constant Linear Velocity.
    - 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.
    - iii. If you select Constant Linear Velocity, enter the desired value.
  - d. Enter a Ramped Flow/Ramped Pressure.

**Note** When you select Ramped Flow or Ramped 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 1600/TRACE 1610 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 159 in Chapter 8, "Split/Splitless Backflush Injector (SSLBKF) Module."

**Note** Select the backflush type (pre-, middle, post-column backflush) on the inlet configuration page.

In **Large volume** mode (only use with the pre-column configuration) 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.
- 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 251 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 31 shows a generic temperature program of the PTV injection cycle.



Figure 31. Generic Temperature Profile

- 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 ramped pressure or ramped 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 1600/1610 Hardware Manual*.



# Flame Ionization Detector (FID) Module

This chapter describes the Instant Connect 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 32. Flame Ionization Detector Module

The module and detector components are shown in Figure 33 and Figure 34.



Figure 33. FID Module Components



Figure 35. 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 9. 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 9. 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 will 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 1600/1610 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 36.



Figure 36. FID Checkout Resulting Chromatogram

The acceptance values in Table 10 indicate successful completion of the checkout. See If these criteria are not met, repeat the test.

 Table 10.
 FID Checkout Acceptance Values

Baseline Parameters	
Noise (fA)	≤ 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 Instant Connect 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 38 and Figure 39.

Figure 38. NPD Module Components



**Figure 39.** 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 1600/1610 Spare Parts Guide*. For replacing a NPD thermionic source refer to the *TRACE 1600/1610 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 11. 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 11.

Gas	Used As:	Range	Typical Condition
Hydrogen	Fuel gas for NPD plasma	0.1-10 mL/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  $H_2$  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 1600/1610 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 41.

Figure 41. NPD Checkout Resulting Chromatogram



The acceptance values in Table 12 indicate successful completion of the checkout. If these criteria are not met, repeat the test.

 Table 12.
 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	<u>≥</u> 1500



# **Thermal Conductivity Detector (TCD) Module**

This chapter describes the Instant Connect 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 42.** Thermal Conductivity Detector Module



The component of module and detector are shown in Figure 43.

### **In-Series Connection TCD Module**

Two modified version of the TCD module are available for the connection in-series with a second detector installed on the TRACE 1600/1610 GC, or to the methanizer in case of the TRACE 1610 Auxiliary Oven.

- The TCD in-series module with Nickel-Iron (Ni-Fe) filament cell is provided with the kit **PN 19070045**.
- The TCD in-series module with **Tungsten-Rhenium** (W-Re) filament cell is provided with the kit **PN 19070046**.

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 44.



#### Figure 44. TCD Module In-Series

**Note** For installing, connecting, and maintaining the TCD in-series module refer to the sections related to the TCD in the *TRACE 1600/1610 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 13 contains information about the thermal conductivity for a variety of gases

Table 13.	Thermal	Conductivity	of Some	Gases
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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
Oxygen	583
Nitrogen	580
Carbon Oxide	540
Argon	406
Carbon Dioxide	343

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 1600/1610 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 46.

Figure 46. TCD Checkout Resulting Chromatogram



The acceptance values in Table 14 and Table 15 indicate successful completion of the checkout. If these criteria are not met, repeat the test.

Baseline Parameters	
Noise (µV)	≤ 12
Drift (µV/h)	≤ 200
Analytical Results	
Dodecane Signal-to-noise ratio	≥ 450
Tetradecane Signal-to-noise ratio	≥ 450
Hexadecane Signal-to-noise ratio	≥ 450

Table 14. TCD with Nickel-Iron (Ni-Fe) Filament Cell Checkout Acceptance Values

**Table 15.** TCD with Tungsten-Rhenium (W-Re) Filament Cell Checkout Acceptance Values

Baseline Parameters	
Noise (µV)	≤ 12
Drift (µV/h)	<u>≤</u> 200
Analytical Results	
Dodecane Signal-to-noise ratio	≥ 100
Tetradecane Signal-to-noise ratio	≥ 100
Hexadecane Signal-to-noise ratio	<u>≥</u> 100

# **Electron Capture Detector (ECD) Module**

This chapter describes the Instant Connect 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 47. Electron Capture Detector Module



The components of the module and detector are shown in Figure 48 and Figure 49.

**Figure 49.** ECD Detector Cell Components



The maximum operating temperature of the ECD is 400 °C.

**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:





## **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** 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 16.

 Table 16.
 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.



• **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 1600/1610 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-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 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. Please see the section "ECD Baseline Optimization." in Chapter 24, "Analytical Troubleshooting."

- 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 50.

Figure 50. ECD Checkout Resulting Chromatogram



The acceptance values in Table 17 indicate successful completion of the checkout. If these criteria are not met, repeat the test.

Table 17. ECD Checkout Acceptance Values

Baseline Parameters	
Noise (Hz)	≤ 10
Drift (Hz/h)	<u>≤</u> 100
Analytical Results	
Lindane Signal-to-noise ratio	≥ 3000
Aldrin Signal-to-noise ratio	≥ 3000



# Flame Photometric Detector (FPD) Module

This chapter describes the Instant Connect 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 51.** Flame Photometric Detector Module

The control module and detector cell assembly components are shown in Figure 52, Figure 53, and Figure 54.





**Note** The signal, excitation voltage, and ignition/heating cables will not be shown in the next illustrations for graphic convenience.



Figure 53. FPD Detector Assembly Components (Front and Back View)

Figure 54. 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 1600/1610 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 55. 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 55. 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 1600/1610 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 18.

Table 18. 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 1600/1610 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 56 or Figure 57.



Figure 56. FPD Checkout Resulting Chromatogram with 394 nm Filter





The acceptance values in Table 19 indicate successful completion of the checkout. If these criteria are not met, repeat the test.

 Table 19.
 FPD Checkout Acceptance Values

Baseline Parameters	394 nm (S) Filter	526 nm (P) Filter
Noise (pA)	<u>&lt;</u> 950	<u>&lt;</u> 1900
Drift (pA/h)	<u>&lt;</u> 1000	<u>&lt;</u> 1000
Analytical Results		
Parathion Methyl Signal-to-noise ratio	≥ 20	≥ 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 58.

Figure 58. 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 1610 Auxiliary Oven.

For further details refer to the chapter **Adding a Module** on the *TRACE 1600/1610 Hardware Manual*.

The module and pulse generator components are shown in Figure 59 and Figure 60.



Figure 59. PDD Module Components



#### Figure 60. 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 1600/1610 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 61.



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 62 shows the pneumatic circuit of the detector system.

Figure 62. 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 1600/1610 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 400 °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 1600/1610 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  $\mu$ L 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 63.

Figure 63. PDD Checkout Resulting Chromatogram



The acceptance values in Table 20 indicate successful completion of the checkout. If these criteria are not met, repeat the test.

#### Table 20. PDD Checkout Acceptance Values

Baseline Parameters	
Noise (pA)	≤ 10
Drift (pA/h)	<u>≤</u> 100
Analytical Results	
Dodecane Signal-to-noise ratio	≥ 500
Tetradecane Signal-to-noise ratio	≥ 500
Hexadecane Signal-to-noise ratio	≥ 500



# **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 1600/1610 GC or on the TRACE 1610 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 320.
- 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 322 for details.

# **GDI Mechanical Module Overview**

Figure 64. GDI Mechanical Module ٠ . Top View 0 6 0 Bottom View Interior View

The GDI mechanical module components are the shown in Figure 64.

The GDI mechanical module components are the shown in Figure 65 and Figure 66.



Figure 65. 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 67.



Figure 67. 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 68.



Figure 68. 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 69.



Figure 69. GDI Electrical Interface

The GDI electrical interface includes the following connections. See Figure 70.





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 1050 kPa (152 psi). The working inlet pressure range is from 400 kPa (58 psig) to 1050 kPa (152 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 1600/1610 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 71.

Figure 71. 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 1600/1610 Hardware Manual* for details.

Two protecting fuses are present inside the GDI electrical interface. See Figure 72 and Table 21.


Figure 72. GDI Electrical Interface Protecting Fuses

Table 21. GDI Electrical Interface Protecting Fuses

Fuse	Туре	Protections
F1	T300 mA 250 V; (5 x 20 mm)	24 Vdc
F2	T2A 250 V; (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**

Figure 73 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 73. 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 74. Analog Output Interface Module







Figure 75. 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, 1 V, 5 V, and 10 V.
- 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 1600/1610 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.

These parameters are configured in the **Configuration** page of the TRACE 1610 user interface. See "Analog Output Interface Menu" on page 45.

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 1610 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 Time Event 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 (SSL-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 76.





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* 1600/1610 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 77.

**Figure 77.** 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 1600/1610 Hardware Manual*.

# Instant Connect Thermospray SSL Injector Module (SSL-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 1600/1610 series GC. See Figure 78.



Figure 78. 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.

# **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 1610**

To confirm that a TRACE 1610 is powered on, after the startup routine, make sure the front touch screen displays the Main Menu. See Figure 79.



Figure 79. TRACE 1610 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 1600**

To confirm that a TRACE 1600 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 80.

Power Start Stop	Ready	Run	Power On Status
Power Start Stop	C Ready	Run	Stand-by Status

Figure 80. TRACE 1600 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 1610 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 1610 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 temperature control. 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 1600/1610 and each stage in a run through the touch screen or the status panel.
  - When the **Not Ready** status is visualized, the TRACE 1600/1610 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 1600/1610 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 1600/1610 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 1600/1610 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 1600/1610 returns to initial temperature and pressure conditions.
- 10. When the Standby status has been reached, the TRACE 1600/1610 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 1600/1610 and each stage in a run through the touch screen or the status panel.
  - When the **Not Ready** status is visualized, the TRACE 1600/1610 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 1600/1610 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 1600/1610 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 1600/1610 returns to initial temperature and pressure conditions.
- 10. When the **Standby** status has been reached, the TRACE 1600/1610 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 22. Troubleshooting Baseline Issues in Your Data (Sheet 1 of 2)

Behavior	Characteristic	Cause	Remedy
		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.
	General	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.
Drifting	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.
	General	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		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.
Irragular shana	~ S-shaped	Excessive column bleed during column temperature programming.	Reduce the upper column temperature. Bake out the column. Install a high temperature column.
irregular shape		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
		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.
	General	Contaminated column.	Condition the column.
		Contaminated detector gas supply.	Check the gas purity and install appropriate filters
Noiso		Defective electrometer.	Replace electrometer.
110126	High-frequency noise	Detector temperature higher than column maximum temperature.	Reduce the detector temperature to the column temperature upper limit.
		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.
		Defective electrometer or amplifier.	Replace the electrometer or amplifier.
Spiking	General	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 22. Troubleshooting Baseline Issues in Your Data (Sheet 2 of 2)

# **Investigating Peak Issues**

 Table 23.
 Troubleshooting Peak Issues in Your Data (Sheet 1 of 3)

Behavior	Characteristic	Cause	Remedy
		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.
Broadening	General	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 Broad ghost peaks	Contaminated carrier gas.	Replace the cylinder or replace the filter.
		Contamination from laboratory glassware.	Ensure the glassware is clean and contamination-free.
		Decomposition of injected sample.	Decrease the injection port temperature. Use the on-column injection technique.
Ghost peaks		Dirty injection solution.	Carry out adequate clean-up of sample prior to injection.
		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
	All	Integrator wires reversed.	Correct the connections.
Negative peaks	Some	Symptom can be normal.	
		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.
	After solvent peak	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.
		Column broken or disconnected.	Check the column and connections.
	At all	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 Iow.	Increase the column/oven temperature. Do not exceed the recommended maximum temperature for the stationary phase.
Tailing		Dirty liner.	Clean or replace the liner.
		Glass wool or inlet liner causing activity.	Replace with fresh silanized wool and a clean inlet liner.
	Samplo poaks	Inlet temperature too low.	Increase the inlet temperature.
	Sample peaks	Poor or obstructed column connections.	Remake the column inlet connection.
		Wrong stationary phase.	Replace the column according to the column manufacturer literature.

#### **Table 23.** Troubleshooting Peak Issues in Your Data (Sheet 2 of 3)

Behavior	Characteristic	Cause	Remedy
		Incorrect column position in inlet.	Reinstall the column.
Tailing (Continued)	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.
	General	Carrier gas flow rate too high.	Reduce the carrier gas flow rate.
		Column deteriorated.	Replace the column.
Unresolved peaks		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 23. Troubleshooting Peak Issues in Your Data (Sheet 3 of 3)

# **Investigating Results Issues**

 Table 24.
 Troubleshooting Results Issues in Your Data (Sheet 1 of 2)

Behavior	Characteristic	Cause	Remedy
		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.
Low reproducibility of	General	Non reproducible sample injection technique.	Evaluate the sample preparation sequences. Compare the results with a series of standard injections.
peaks area		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.
Poor sensitivity	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.
		Leaks in the GC carrier gas line.	Run a leak test and correct leaks.
	With normal retention time	Syringe leaks during injection.	Replace syringe or piston seals, if applicable.
		Split injection temperature too low.	Increase the temperature of the injector.

Behavior	Characteristic	Cause	Remedy
	Decreasing	Stationary phase deteriorated by oxygen and/or water.	Use a carrier gas free of oxygen and water.
		Stationary phase loss due to column bleeding.	Reduce the column temperature.
	Increasing	Increasing carrier leakage.	Check the septum and column connections.
		Carrier gas supply running out.	Replace the bottle.
Retention times	Low reproducibility	Drifting or unstable pneumatic controller.	Monitor the column pressure or flow. Check and replace the controller if necessary.
		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 24.
 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.
Heavy compounds are not eliminated.	Backflush starts too late due to Transfer time too long.	Reduce transfer time duration.
	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 Large Volume run.	Backflush not enabled Incorrect transfer time or evaporation time duration.	Enable Backflush. Set sufficient time duration.
	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.

#### **ECD Baseline Optimization**

Optimization of the baseline is a very important process that needs to be performed before using the ECD. A too low baseline level will degrade significantly the performances of the ECD, therefore an optimization procedure must be performed every time a new ECD is installed on a TRACE 1600 Series GC.

The optimal ECD baseline level sits in the interval 1 - 1.5 kHz.

- If the ECD baseline level is below 1 kHz, the baseline will become unstable and the noise will increase suddenly and dramatically. This typically happens when the ECD is very clean, e.g. a new ECD.
- If the ECD baseline is above 1.5 kHz, the baseline noise will increase, the more the baseline is above 1.5 kHz, the higher will be the baseline noise. This might happen if the ECD becomes dirty, for example when it is used to analyze dirty matrices that might leave deposits inside the ECD cell.

#### Parameters that Affect the Baseline Level of the ECD

Parameters that affect the baseline level of the ECD are the following:

- **Reference current** This is the current the detector cell is required to produce by collecting the ions generated. It is recommended that **this parameter is never changed**.
- **Pulse amplitude** This is the energy with which the free electrons are pushed against the collector. Higher pulse amplitude means higher energy for collecting electrons and therefore lower baseline, while lower pulse amplitude means higher baseline.
- **Pulse width** Pulse width is the time window of the electron collection voltage pulse applied to the cell. Larger pulse width means longer collection time for each pulse, therefore lower baseline while narrower pulse width means shorter collection time and higher baseline.
- Makeup flow The makeup is the gas that helps the free electrons inside the cell to reduce their energy and be ready for capture. Low makeup gas flow means lower gas purity inside the cell therefore higher baseline. High makeup flow conversely means lower baseline level.
While all these parameters are part of the method settings, to optimize the ECD baseline level it is strongly suggested to act only on Pulse width.

In no case the Reference current shall be set to a value different from the default value of 0.5 nA.

The Pulse Width can be reduced from 1 us (default value for Nitrogen make-up) down to  $0.1 \,\mu$ s, the minimum value of the Pulse Width.

When using  $Ar/CH_4$  as a makeup gas, the suggested initial setting for the Pulse Width is already the minimum value of 0.1 µs.

If, when using  $Ar/CH_4$ , the baseline level becomes lower than 1 kHz, it can be increased by reducing the Pulse Amplitude.

#### **Default ECD Conditions**

When installing a new ECD, to set the initial conditions for the ECD, use the ECD SOP conditions (default setting in the GC and data system user interface).

While these initial conditions are in general a good starting point, a new ECD with a very clean cell might experience very low baseline level and therefore in the majority of the cases needs optimization to increase the baseline level above the minimum limit of 1 kHz.

#### **Optimization of the ECD Baseline Level**

To verify if the ECD baseline level needs optimization, a baseline acquisition is needed.

Acquire a baseline using the default SOP method conditions. If the baseline shows an unusually high noise:



or a noise that is erratic:



It means that the ECD needs optimization. In this example, the baseline level of the ECD is very low, less than 0.725 kHz. It can also be seen that as soon as the baseline drops below 0.675 kHz, the noise dramatically increases and the ECD performances are greatly affected.

To bring the ECD back to its optimal working conditions, we need to increase the baseline level in order to stay well higher than the limit value of 0.675 kHz.

It must be noted that this limit value is not always exactly the same: this is why the ECD baseline level must be raised over the safe value of 1 kHz.

To raise the ECD baseline level we have the mentioned ECD method parameters to work with, but the greatly preferred parameter is the Pulse Width.

Reduce the Pulse Width until the unusual noise on the baseline disappears and the baseline level stabilizes above 1 kHz, in the range 1-1.5 kHz.

After optimization, the baseline looks like:



With a nice low noise and very sensitive ECD. Now the ECD is working at its best performances.

With time the ECD might become contaminated with the sample matrix: in this case its baseline level will increase. To decrease it, restoring the ECD optimal conditions, it is enough to increase back the Pulse Width.

# **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 Check connections								
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 367.

- 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 367.

- 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 365.

#### 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 367.
- 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	Χ	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

**CPU** central processing unit (of a computer)

<Ctrl> control key of the keyboard

#### 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 •F Fahrenheit

FID Flame Ionization Detector

FOB Free on board

FSE Field Service Engineer

FPD Flame Photometric Detector

G

#### 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 ( $10^3$  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

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