Thermo Scientific

TRACE GC Ultra

Gas Chromatograph

Operating Manual

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Home

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- installation
- re-calibration
- changes and repairs

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- 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

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About This Manual

Overview

This *Operating Manual* contains descriptions of the features and components of the TRACE GC Ultra gas chromatograph. Inside, you will find all of the information necessary for routine operation of your GC, including operating sequences, sample injection techniques, and diagrams and descriptions of the major components.

This manual is organized as follows:

Section I familiarizes you with your TRACE GC Ultra gas chromatograph. In addition to basic descriptions of TRACE GC Ultra features and systems, this section contains instructions for configuring and interacting with your GC.

Chapter 1, *TRACE GC Ultra Overview*, provides a basic overview of the features and options of the TRACE GC Ultra gas chromatograph.

Chapter 2, *The TRACE GC Ultra User Interface*, gives a general overview of the TRACE GC Ultra user interface, including basic information about key functions and menus.

Chapter 3, *Configuration*, describes how to set up the software on your TRACE GC Ultra either to match the installed hardware or to reflect your preferences.

Section II contains information on controlling and programming the detector and carrier gas flows to the TRACE GC Ultra.

Chapter 4, *Digital Gas Control*, This chapter describes the automatic (DCC and DGFC) gas control features of the TRACE GC Ultra and contains the instructions to program and regulate the GC carrier gases control.

Section III contains information about the injection systems available for the TRACE GC Ultra.

Chapter 5, *Split/Splitless Injector (S/SL)*, describes the Split/Splitless (S/SL) injector and contains operating sequences for the different split/splitless operating modes.

Chapter 6, *On-Column Injector (OCI)*, describes the On-Column injector (OCI), on-column injection techniques, and operating sequences.

Chapter 7, *High Oven Temperature Cold On-Column Injector (HOT OC)*, describes the HOT Cold On-Column (HOT OC) injector for injections at high oven temperatures, HOT on-column injection techniques, and operating sequences.

Chapter 8, *Large Volume On-Column Injector (LVOCI)*, describes the Large Volume On-Column Injector (LVOCI) used for large volume injections with an autosampler.

Chapter 9, *Packed Column Injector (PKD)*, describes the Packed (PKD) column injector and explains the packed column operating sequences.

Chapter 10, *Purged Packed Column Injector (PPKD)*, describes Purged Packed Column (PPKD) injector, which has a septum purge option. Included in this chapter are PPKD injection techniques and operating sequences.

Chapter 11, *Programmable Temperature Vaporizing Injector (PTV)*, describes the Programmable Temperature Vaporizing (PTV) injector and contains operating sequences for using the injector in different operating modes.

Chapter 12, *Gas Sampling Valve (GSV)*, describes the gas sample valves available with the TRACE GC Ultra and contains operating sequences for manual and automatic sampling.

Section IV contains information about the configuration options for the TRACE GC Ultra column oven and sequences for using capillary and packed columns in the oven.

Chapter 13, *The Column Oven*, describes the features and configuration options for the TRACE GC Ultra column oven and includes operating sequences for oven programming.

Chapter 14, *Columns*, describes the analytical columns used in the TRACE GC Ultra.

Section V contains information about detector configuration and operation.

Chapter 15, *Detector Overview*, gives basic information about the detectors available with the TRACE GC Ultra.

Chapter 16, *Flame Ionization Detector (FID)*, describes the operating principles and sequences for the Flame Ionization Detector (FID).

Chapter 17, *Electron Capture Detector (ECD)*, describes the operating principles and sequences for the Electron Capture Detector (ECD).

Chapter 18, *Nitrogen Phosphorus Detector (NPD)*, describes the operating principles and sequences for the Nitrogen Phosphorus Detector (NPD).

Chapter 19, *Photoionization Detector (PID)*, describes the operating principles and sequences for the Photoionization Detector (PID).

Chapter 20, *Flame Photometric Detector (FPD)*, describes the operating principles and sequences for the Flame Photometric Detector (FPD).

Chapter 21, *Thermal Conductivity Detector (TCD)*, describes the operating principles and sequences for the Thermal Conductivity Detector (TCD).

Chapter 22, *Pulsed Discharge Detector (PDD)*, describes the operating principles and sequences for the Pulsed Discharge Detector (PDD).

Section VI contains informations about AI 3000 / AS 3000 programming with the TRACE GC Ultra keypad.

Chapter 23, *AI 3000 / AS 3000 Autosampler*, describes how to program and control the AI 3000 / AS 3000 autosampler by using the TRACE GC Ultra keypad.

Section VII contains descriptions of automated and manual control options and sequences for the TRACE GC Ultra.

Chapter 24, *Automated Functions*, shows you how to automate signal, valves, and external events by scheduling them either in real time (clock table events) or at certain points during a run (run table events). It also discusses the run log, an automated record of run deviations.

Chapter 25, *Manual Functions*, describes how to control signal and valve events manually.

Section VIII contains information on programming analytical methods and using them in autosampler injection sequences.

Chapter 26, *Using Analytical Methods*, describes how to set up analytical methods that run automatically when specified.

Chapter 27, *AI 3000 / AS 3000 Autosampler Sequences*, contains the instructions to programming a sample sequence with the TRACE GC Ultra

keypad when an AI 3000 / AS 3000 autosampler is used and how to set up ranges of samples to run automatically.

Appendix A, *Ionization Potential of Selected Molecules*, contains information to help you determine the PID lamp intensity necessary to ionize certain molecules.

Appendix B, *Customer Communication*, contains contact information for Thermo Fisher Scientific offices worldwide. This appendix also contains a one-page *Reader Survey*.

The *Glossary* contains definitions of terms used in this guide and the help diskette. It also includes abbreviations, acronyms, metric prefixes, and symbols.

The *Index* contains an alphabetical list of key terms and topics in this guide, including cross references and the corresponding page numbers.

Conventions Used in This Manual

The following symbols and typographical conventions are used throughout this manual.

Bold	Bold text indicates names of windows, dialog boxes, and fields.
Italic	Italic indicates cross references, first references to important terms defined in the glossary, and special emphasis.
`Monospace`	Monospace, or Courier, indicates filenames and filepaths or text the user should enter with the keyboard.
`Monospace Bold`	Monospace Bold indicates messages, prompts, or menu titles displayed on the computer screen or on a digital display.
»	This symbol illustrates menu paths to select, such as **File»Open…**.
KEY NAME	Bold, uppercase sans serif font indicates the name of a key on a keyboard or keypad, such as ENTER.

This symbol alerts you to an action or sequence that, if performed improperly, could damage the instrument.

This symbol alerts you to important information related to the text in the previous paragraph.

This symbol alerts you to an action or sequence that, if improperly performed, could result in damage to the instrument or possible physical harm to the user. This symbol may be followed by icons indicating special precautions that should be taken to avoid injury.

This symbol indicates an electric shock hazard.

This symbol indicates danger from hazardous chemicals.

This symbol indicates danger from high temperature surfaces or substances.

This symbol indicates a fire hazard.

This symbol indicates an explosion hazard.

This symbol indicates a toxic hazard.

This symbol indicates the presence of flammable materials.

This symbol indicates the presence of radioactive material.

This symbol indicates an operation or sequence that must *not* be performed by the user. A Thermo Fisher Scientific authorized Customer Support Engineer must perform this sequence.

This symbol indicates all metal objects, such as watches and jewelry, must be taken off.

This symbol indicates an eye hazard. Eye protection must be worn.

This symbol indicates the user must wear a protective screen when performing the sequence.

This symbol indicates the user must wear protective shoes when performing the sequence.

This symbol indicates the user must wear protective clothing when performing the sequence.

This symbol indicates the user must wear gloves when performing the sequence.

Instrument Markings and Symbols

The following table explains the symbols used on Thermo Fisher Scientific instruments. Only a few of them are used on the TRACE GC Ultra gas chromatograph.

Symbol	Description
	Direct Current
	Alternating Current
	Both direct and alternating current
	Three-phase alternating current
	Earth (ground) terminal
	Protective conductor terminal
	Frame or chassis terminal
	Equipotentiality
	On (Supply)
	Off (Supply)

Symbol	Description
	Equipment protected throughout by DOUBLE INSULATION or REINFORCED INSULATION (Equivalent to Class II of IEC 536)
	Indicates that the user must refer to the manual for specific Warning or Caution information to avoid personal injury or damage to the product.
	Caution, risk of electric shock
	Caution, hot surface
	Caution (refer to accompanying documents)
	In-position of a bistable push control
	Out-position of a bistable push control
	Symbol in compliance to the Directive 2002/96/EC on Waste Electrical and Electronic Equipment (WEEE) placed on the european market after August, 13, 2005.

Using the TRACE GC Ultra Document Set

The TRACE GC Ultra Document Set (CD-ROM PN 317 095 00) includes all manuals in electronic format, and serves as your library for information about the TRACE hardware and software.
The TRACE GC Ultra Document Set (PN 317 093 00) as paper copy is also available Furthermore, Thermo Fisher Scientific part numbers (PN) for the paper copy manuals are provided for each book title.

Site Preparation and Installation Manual (PN 317 091 90)
This manual and diskette describes how to set up a workspace for the TRACE GC and how to connect the TRACE GC Ultra to the gas supplies and peripheral devices.

Acceptance Package (PN 317 092 20)
This folder contains required shipping documents and quality report forms.

Getting Started (PN 317 092 30)
This guide contains sequences for checking configuration, installing detectors, and making a first analysis with the TRACE GC Ultra.

Operating Manual (PN 317 091 70)
This manual provides descriptions of the TRACE GC Ultra hardware and software and instructions for their use.

UFM Ultra Fast Module Device (PN 317 093 98)
This manual provides descriptions of the TRACE GC Ultra equipped with the UFM device. and instructions for it use.

Quick Reference Card (PN 317 092 40)
This reference card contains guidelines for carrier gas use and injection sequences.

K-Factor Quick Reference (P/N 317 092 41)
This card indicates the theoretical K-Factor related to the carrier gas and the column in use.

Preventive Maintenance Schedule (PN 317 092 80)
This document provides a list of recommended scheduled maintenance and a year-long log book to record maintenance, observations, supply lists, and service records.

Maintenance and Troubleshooting Guide (PN 317 091 80)
This manual contains instructions for diagnosing and resolving operational problems.

Standard Operating Procedures (PN 317 092 00)
This manual contains instructions, operating sequences, and test criteria for final testing of the TRACE GC Ultra.

Spare Parts Catalog (PN 317 092 10)
This catalog contains a list of spare parts for the TRACE GC Ultra.

Using Hydrogen

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

CAUTION! Hydrogen is not compatible with the MS detector.

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

Use the following safety precautions when using hydrogen:

- Ensure that all hydrogen cylinders comply with the safety requirements for proper use and storage. Hydrogen cylinders and delivery systems must comply with local regulations.
- Make sure the gas supply is turned completely off when connecting hydrogen lines.
- Perform a bubble test to ensure that the hydrogen lines are leak-tight before using the instrument. Perform the bubble test after performing the pressure test described in the *TRACE GC Ultra Maintenance and Troubleshooting Manua*l.
- Avoid spraying any electrical components during the bubble test. Continue checking each section of the pneumatic circuit until you identify the leak. If you need to perform a leak check inside the pneumatic compartment, first perform the bubble test with all circuits pressurized, then disconnect the GC from the main gas supply and remove the pneumatic circuit panel. Repeat this sequence until you eliminate all leaks.
- Ensure your GC column oven has a hydrogen sensor. A hydrogen sensor continuously monitors the hydrogen level in the GC column oven.

  If your GC oven does not have a hydrogen sensor already installed, contact your Thermo Fisher Scientific sales representative. To comply with instrument safety requirements, a Thermo Fisher Scientific CSE or authorized service technician should install the sensor.

  If you plan to use a sensor other than the sensor recommended by Thermo Fisher Scientific, you must verify its ability to perform the functions listed above before installing it. It must comply with your local safety regulations, or with the IEC 61010[1] regulations if local regulations do not exist.

Using the Hydrogen Sensor

The lower limit of the hydrogen sensor is 0.5% in volume. You should adjust the detection threshold to 1% in volume, which is 25% of the hydrogen lower limit of explosion (4% in volume).

1. IEC 1010-1, First Edition, September 1990; IEC 1010-1, Amendment 1, September 1992; IEC 1010-1, Amendment 2, June 1995.

In cases where the connections begin to leak or the column breaks, the sensor alerts the operator. It then automatically cuts off the gas supply and heating to the active zones, and sweeps the column oven with forced air ventilation.

If the sensor detects anomalies or leaks during GC operation due to instrument malfunction, the operator must immediately:

- close the hydrogen supply
- switch off the gas chromatograph
- air out the room

The reliability of the sensor depends on careful maintenance. After 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.

WARNING! Never use hydrogen in your TRACE GC Ultra system unless your GC oven has a hydrogen sensor installed.

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

Using Liquid Coolants

High pressures and extremely low temperatures make pressurized liquid CO_2 and liquid N_2 hazardous materials.

- High concentrations of CO_2 are dangerous.
- High concentrations of N_2 in the air can cause an asphyxiation hazard.

To avoid injury, always follow the safety precautions and delivery system design recommended by your gas supplier.

SECTION

I

TRACE GC Ultra Basics

This section familiarizes you with your TRACE GC Ultra gas chromatograph. In addition to basic descriptions of TRACE GC Ultra features and systems, this section contains instructions for configuring and interacting with your GC.

Chapter 1, *TRACE GC Ultra Overview*, provides a basic overview of the features and options of the TRACE GC Ultra gas chromatograph.

Chapter 2, *The TRACE GC Ultra User Interface*, gives a general overview of the TRACE GC Ultra user interface, including basic information about key functions and parameter tables.

Chapter 3, *Configuration*, describes how to set up the software on your TRACE GC Ultra either to match the installed hardware or to reflect your preferences.

1

TRACE GC Ultra Overview

This chapter provides a basic overview of the features and options of the TRACE GC Ultra gas chromatograph. After each brief description of a TRACE GC Ultra component, you will find references to chapters in this manual containing more detailed information.

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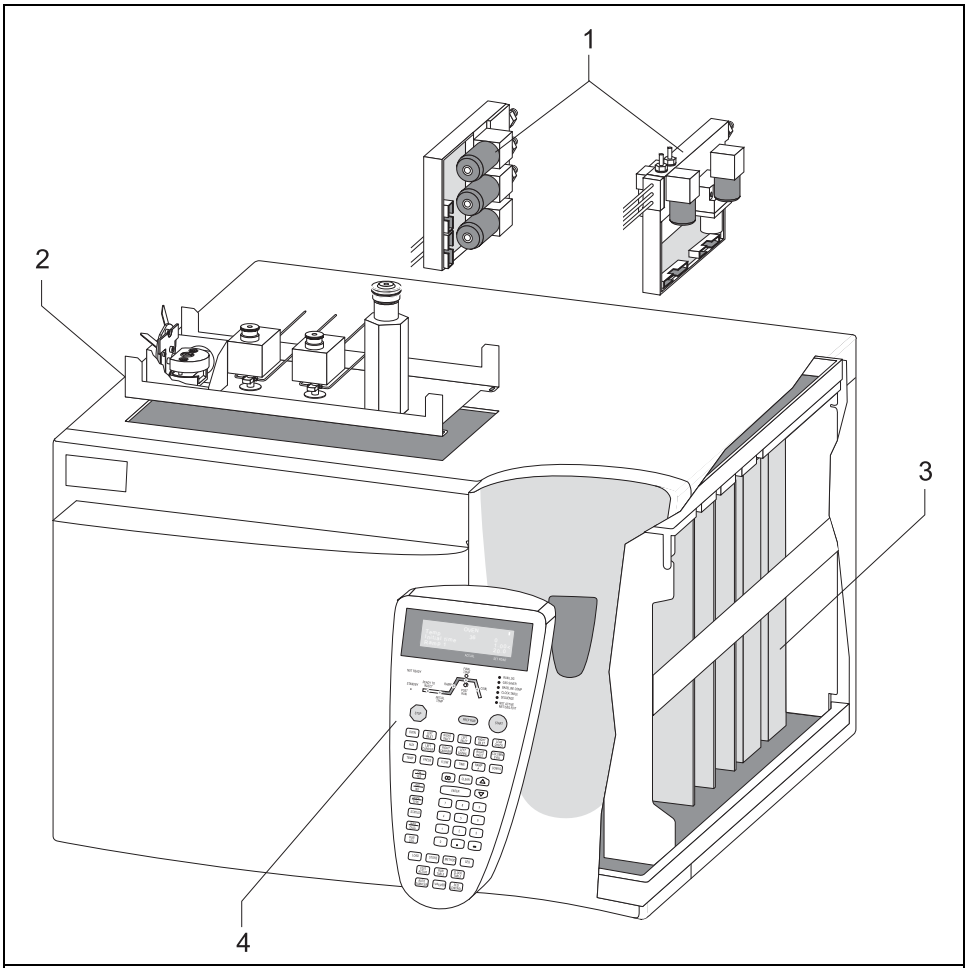
TRACE GC Ultra System Components

The TRACE GC Ultra consists of four major components, as shown in Figure 1-1.



1. Pneumatic Compartment	3. Electronic Compartment
2. Analytical Unit	4. Display and Keyboard

Figure 1-1. TRACE GC Ultra Components

Pneumatic Compartment

The pneumatic compartment contains the pneumatic gas control circuits.

Analytical Unit

The analytical unit consists of two subcompartments:

- the column oven
- the injector and detector compartment

Electronic Compartment

The electronic compartment consists of two subcompartments:

- the high-voltage compartment
- the motherboard for the detector control cards

Display and Keypad

The display and the keypad make up the TRACE GC Ultra user interface.

Cleaning and Decontamination

Normal usage of the TRACE GC Ultra can cause the exterior to get dirty. Clean the outer surfaces by wiping them with a cloth dampened with water.

In the event that a hazardous material is spilled on or in the instrument, clean the spill according to the procedures in the Material Safety Data Sheet for that substance.

Gas Control

The arrangement of the pneumatic gas control system depends on the detectors configured on the base unit. Digital Pneumatics.
Carrier and detector gases are controlled electronically through a series of electronic pneumatic control modules mounted in the pneumatic compartment. The Digital Carrier Control (DCC) modules control the carrier gas flow and the Detector Gas Flow Control (DGFC) modules control the detector gas flow.

A single DCC module can alternate the flow of one carrier gas supply between a split/splitless injector and another (non-split/splitless) injector.

Carrier Gas Control

The DCC module allows the digital control of the inlet pressure and carrier gas flow and features the following:

- constant pressure or constant flow operating modes
- programmed pressure or programmed flow operating modes
- inlet pressure control (in kPa, psi, or bar) and column flow rate control (in mL/min)
- split flow control (in mL/min)
- septum purge flow control (in mL/min)

The DCC module also allows the following operations:

— **Column Evaluation**
To calibrate the DCC module according to the real carrier flow rate.

— **Leak Check**
To assure the tightness of the system.

— **Gas Saver Function**
To reduce the split flow after an injection to avoid the waste of expensive gases.

There are three types of DCC modules:

- for OCI and PKD injectors

- for PPKD injector
- for S/SL and PTV injectors

Refer to...
Chapter 4, *Digital Gas Control*

Detector Gas Control

The DGFC module allows the digital control of all the necessary detector gases. The DGFC can be automatically switched on and off using the TRACE GC Ultra keypad.

They are four types of DGFC modules

- for ECD only (Type AA)
- for ECD, PID, FPD, FID without make-up gas (Type AB)
- for ECD, PID, FPD, FID with make-up gas (Type AC)
- for NPD, ECD, PID, FPD, FID without make-up gas (Type AD)

Refer to...
Chapter 4, *Digital Gas Control*; and Chapter 15 *Detector Overview*.

Injectors

The following injectors are available on the TRACE GC Ultra:

- Split Splitless Injector (S/SL and LVSL)
- On-Column Injector (OCI)
- HOT Cold On-Column Injector (HOT OC)
- Large Volume On-Column Injector (LVOCI)
- Packed Column Injector (PKD)
- Packed Column Injector with Septum Purge (PPKD)
- Programmable Temperature Vaporizing Injector (PTV and PTVLVI)
- Gas Sampling Valves (GSV)

Split Splitless Injector

The Split/Splitless (S/SL, LVSL) injector minimizes heavy component discrimination with optimized sample transfer to the column. You can use capillary and wide-bore columns with the Split/Splitless injector. With the appropriate adapter kit, you can also use packed columns.

Refer to...
Chapter 5, *Split/Splitless Injector (S/SL)*

On-Column Injector

The On-Column Injector (OCI) allows you to inject a sample directly into a 0.25 or 0.32 mm capillary column and 0.53 mm wide-bore column. Primary and secondary cooling systems keep the injection block at ambient temperature and the injection zone cool to prevent sample vaporization and ensure complete sample transfer from the syringe to the column.

Refer to...
Chapter 6, *On-Column Injector (OCI)*

HOT Cold On-Column Injector

The High Oven Temperature Cold On-Column (HOT OC) injector is a special version of the standard on-column injector. It use an optional HOT device to operate at high oven temperatures.

Refer to...
Chapter 7, *High Oven Temperature Cold On-Column Injector (HOT OC)*

Large Volume On-Column Injector

The Large Volume On-Column Injector (LVOCI) is a special version of the standard on-column injector. It allows automatic introduction of large volumes of liquid sample through the TriPlus AS autosampler.

Refer to...
Chapter 8, *Large Volume On-Column Injector (LVOCI)*

Packed Column Injector

The Packed Column (PKD) injector allows injection directly into metal or glass packed columns or into metal or glass packed columns with glass liners.

Refer to...
Chapter 9, *Packed Column Injector (PKD)*

Purged Packed Column Injector

The Purged Packed Column (PPKD) injector allows sample injection and vaporization into a liner. The sample then transfers to a wide-bore capillary column.

Refer to...
Chapter 10, *Purged Packed Column Injector (PPKD)*

Programmable Temperature Vaporizing Injector

The Programmable Temperature Vaporizing (PTV, PTVLVI) injector allows temperature variation during the injection process in both split and splitless operating modes.

Refer to…
Chapter 11, *Programmable Temperature Vaporizing Injector (PTV)*

Gas Sampling Valves

Two gas sampling valves for manual and automatic sampling are available with the TRACE GC Ultra. It allows manual and automatic gas sampling.

Refer to…
Chapter 12, *Gas Sampling Valve (GSV)*

Column Oven

The GC column oven has a high degree of thermal stability and fast heating and cooling. The air circulation in the oven ensures the column is kept in a thermally homogenous and stable zone. This provides more precise analytical performance and helps prevent chromatogram peak distortion.
The oven can operate at temperatures below ambient with a cryogenic cooling system. The cryogenic system allows oven temperatures down to –55 °C with liquid carbon dioxide or –99 °C with liquid nitrogen.

Refer To…
Chapter 13, *The Column Oven*

Ultra Fast Module Device

The Ultra Fast Module (UFM) device includes a column module where a capillary column, a heating wire and a temperature sensing wire are combined in a tight package using a ceramic fiber insulation.

Compared to conventional air circulating GC oven, UFM Device features faster temperature programming and it allows heating rates up to 1200 °C/min while maintaining moderate power consumption. The device control is performed by a dedicated control card installed in the GC electronic compartment.

NOTE

The UFM Device is automatically recognized when the GC is turned on and a relevant message is displayed during the GC start-up routine.

Refer To…
UFM Ultra Fast Module Device Instruction Manual

Columns

The column is where the chromatographic separation of the sample occurs. Several types of columns are available for different chromatographic applications:

- capillary columns
- wide-bore capillary columns
- metal packed columns
- glass packed columns

Refer to…
Chapter 14, *Columns*

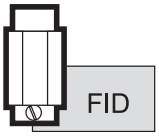
Detectors

The following detection systems are available for the TRACE GC Ultra:

- Flame Ionization Detector (FID)
- Electron Capture Detector (ECD)
- Nitrogen Phosphorus Detector (NPD)
- Photoionization Detector (PID)
- Flame Photometric Detector (FPD) [*Single and Dual Configurations*]

- Thermal Conductivity Detector (TCD)
- Pulsed Discharge Detector (PDD)

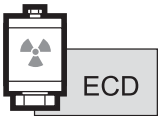
Flame Ionization Detector



The Flame Ionization Detector (FID) is one of the most useful detectors in GC because of its high sensitivity, good stability and wide range of linearity of response. The FID ensures stable, reproducible, and long-term trouble-free performance.

Refer to…
Chapter 16, *Flame Ionization Detector (FID)*

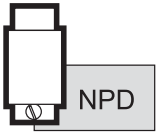
Electron Capture Detector



The Electron Capture Detector (ECD) is a non-destructive detector that utilizes the ability of many compounds to capture electrons. It features a very low ionization cell volume and increased resistance to contamination. This ensures high sensitivity and trouble-free operations. You can easily remove and clean the collecting electrode without disturbing the ^{63}Ni source.
The detector can be heated to 400 °C, extending its application range to higher molecular weight compounds.

Refer to…
Chapter 17, *Electron Capture Detector (ECD)*

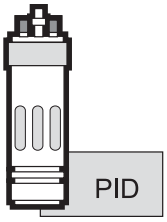
Nitrogen Phosphorus Detector



The Nitrogen Phosphorus Detector (NPD), equipped with a ceramic matrix thermionic source, features high sensitivity and long-term stability for analyzing compounds containing nitrogen and phosphorus. A special thermionic source is also available for Enhanced Nitrogen Selectivity (ENS) mode.

Refer to…
Chapter 18, *Nitrogen Phosphorus Detector (NPD)*

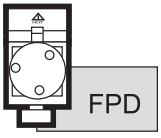
Photoionization Detector



The Photoionization Detector (PID) is mainly used to determine aromatic pollulant compounds in environmental applications and to analyze policyclic aromatic hydrocarbons. It uses a UV lamp to energize the sample eluted from the chromatographic column. The type of lamp used determines the selectivity and sensitivity of the detector. The PID is widely used in the environmental field to test for aromatic and policyclic hydrocarbons.

Refer to…
Chapter 19, *Photoionization Detector (PID)*

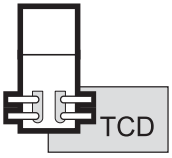
Flame Photometric Detector



The Flame Photometric Detector (FPD) is based on the emission photometric principle. It is one of the most selective detectors in gas chromatography. The high sensitivity and good linear dynamic range (log scale for sulphur response) provide excellent performance for trace determination of sulphur and phosphorus containing compounds. Some uses of the FPD include pesticide residue analysis, pollution control, and crude oil analysis.
This detector may also operate in Dual FPD Configuration (Twin tube) installing a second photomultiplier tube available as option in the relevant upgrade kit.

Refer to…
Chapter 20, *Flame Photometric Detector (FPD)*

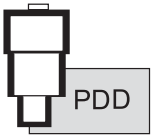
Thermal Conductivity Detector



The Thermal Conductivity Detector (TCD) is a dual filament, single column detector. Its response depends on the difference between the thermal conductivity of pure carrier gas and that of carrier gas containing eluted sample. The TCD features output signal amplification by a factor of 10. Two operating control modes are possible: *constant temperature*, which ensures a high degree of filament protection and high sensitivity, and *constant voltage*, which extends the linear dynamic range to greater than 10^5.

Refer to…
Chapter 21, *Thermal Conductivity Detector (TCD)*

Pulsed Discharge Detector



The Pulsed Discharge Detector (PDD) is an universal and highly sensitive non-radioactive and nondestructive 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.

Refer to…
Chapter 22, *Pulsed Discharge Detector (PDD)*

Multidetector System

You can use a multidetector configuration to significantly reduce analysis time and increase analytical information for complex samples providing a number of chromatograms from each single injection. Detectors may be arranged:

- **in series**
  with a non-destructive detector (ECD, PID, TCD) followed by a destructive detector (NPD, FPD or FID).
- **in parallel**
  by using an effluent splitter for fused silica capillary column.
  This may be particularly useful for bulk analysis of product formulations, biochemical, and environmental applications.

Detector Base Bodies

The ionization detectors are easily interchangeable. This is made possible by *base bodies* on the analytical unit that provide a connection between the detector and the analytical column.
Two types of detector base bodies are available:

- for packed column
- for capillary column

The type you can use depends on the GC base unit configuration.

Refer to…
Chapter 15, *Detector Overview*

Instrument Automation

The TRACE GC Ultra contains several automated features for running the GC, communicating with other analysis equipment, and interacting with a data system.

Internal Automation

You program internal automation by entering run time and real-time clock events in special menus. You can set these events to execute at specified times after injection, at specified times during the day, and on specific days of the week.

Refer to…
Chapter 24, *Automated Functions*

Communication with External Units

You can connect the TRACE GC Ultra to external modules and accessories, such as data systems, autosamplers, and mass spectrometers.

Autosampler Interface

TriPlus and AI 3000/AS 3000 autosamplers can be connected to the GC.

Refer to…
Chapter 23, *AI 3000 / AS 3000 Autosampler*
TriPlus Operating Manual

Data Systems Interface

Your TRACE GC Ultra generates analog and digital data output when you perform chromatographic analysis. A computer with a Thermo Scientific data system can be used to process the data from the GC.

According to the CPU board, standard or LAN, installed into the GC, the communication between the data system and the instrument is performed through RS232 serial line and Local Area Network respectively.

Methods and Sequences

You can program analytical methods and sequences for autosamplers in the TRACE GC Ultra menus.

Sequences tell the autosampler where the samples are located in the autosampler tray and the order in which to analyze them. Methods control the analysis parameters used during a sequence. You can store up to ten methods and five sequences in memory.

Refer to…
Chapter 26, *Using Analytical Methods*
Chapter 27, *AI 3000 / AS 3000 Autosampler Sequences*

2

The TRACE GC Ultra User Interface

This chapter gives a general overview of the TRACE GC Ultra user interface, including basic information about key functions and menus.

The TRACE GC Ultra gas chromatograph is often used with a data system and external devices, such as an autosampler. However, most functions can be programmed through the GC.

The user interface has three components: a four-line display, display LEDs showing the instrument's status, and a keypad for data entry.
Each component is discussed in order from the top down. Figure 2-1 illustrates the complete TRACE GC Ultra user interface.

Chapter at a Glance…

The Display 49
The Display LEDs 50
The TRACE GC Ultra Keypad 53
General Navigation 66
Error Conditions 68

Operating Sequences

Editing a Menu Item 67

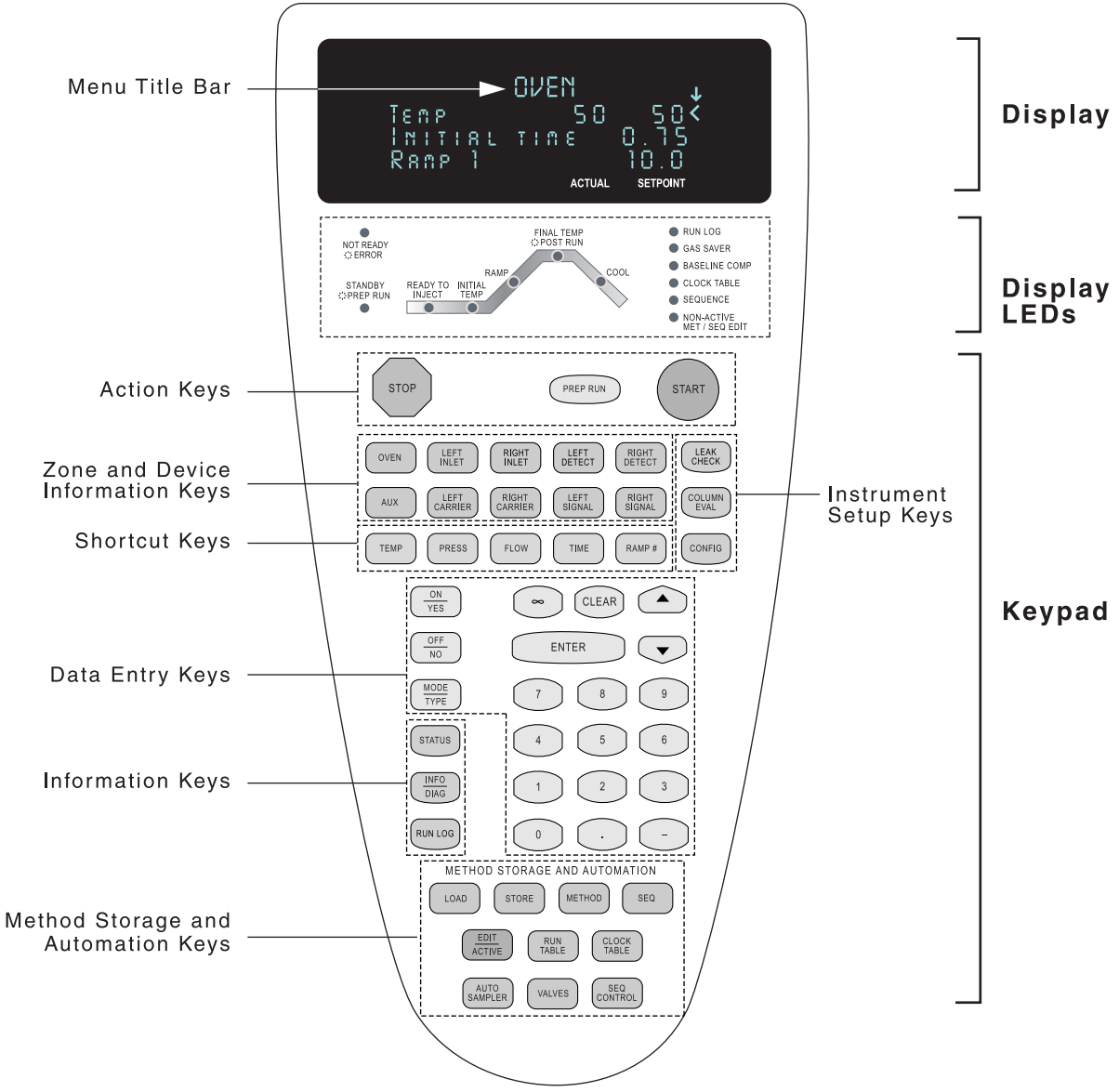


Figure 2-1. The TRACE GC Ultra User Interface

The Display

The display shows the menus you use to control the GC parameters, settings, and configuration options. To open a menu, press its associated key. For example, press the **LEFT INLET** key to open the **LEFT INLET** menu. The data entry keys allow you to scroll through, set, and modify the menu information.

Figure 2-2 shows the components of a typical menu display.

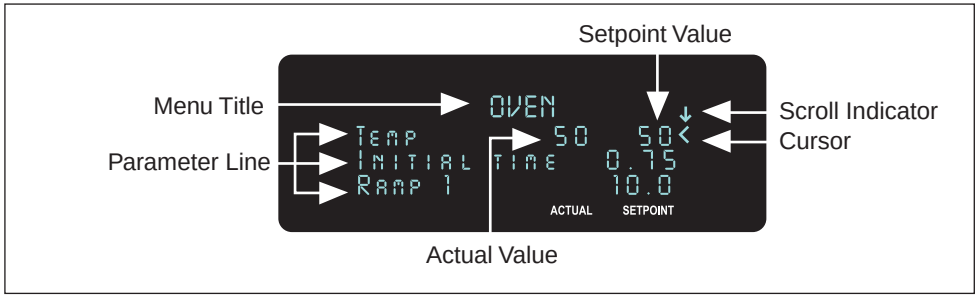


Figure 2-2. Components of the TRACE GC Ultra Menu Display

The following are the menu display components:

Menu Title—This is the first line of each menu. The menu title remains at the top of the display and does not move, even when you scroll up and down the menu items.

Cursor—The cursor indicates the currently selected menu item. Use the **UP ARROW** and **DOWN ARROW** keys to move the cursor.

Setpoint Value and *Actual Value*—Many parameters display two values. The first value is the actual value of the GC parameter. You enter the second value, which is the setpoint.

Scroll Indicator—This item is found in the upper right corner of the display. It indicates when not-currently visible menu items exist. It appears in three ways:

- ↓, indicating that you can scroll downward
- ↑, indicating that you can scroll upward

- ↕, indicating that you can scroll in either direction

Currently Visible Menu Parameters—The display shows four lines of a menu at a time. Because the menu title always takes up the first line, three lines show menu items.

Not Currently Visible Menu Parameters—The display shows three menu items at a time. If a menu contains more than three lines, you can use the arrow keys to scroll through the rest of the menu items.

The Display LEDs

The LEDs (Light Emitting Diodes) below the display screen indicate the TRACE GC Ultra's operating status.

The Status LEDs

The status LEDs indicate the current operating mode and special settings activated by the operator. Table 2-1 lists and explains each status LED.

Table 2-1. Status LED Descriptions

LED	Description
Not Ready/Error	This LED lights when the GC is not ready to make a run, usually because the specified oven temperature has not been reached. It remains lit if any additional equilibration time has been configured. It blinks when the GC has one or more error conditions.
Standby/Prep Run	This LED lights when the GC is in **Standby**, waiting to be advanced to the **Ready** status. It blinks links while the GC prepares for a run, for example, while opening or closing valves as required by the method or waiting for an external device such as a mass spectrometer.
Run Log	This LED lights when the GC records a run error or a parameter changes during a run.
Gas Saver	This LED lights when the gas saver function is enabled.
Baseline Comp	This LED lights when baseline compensation is used.

Table 2-1. Status LED Descriptions (Continued)

LED	Description
Clock Table	This LED lights when the clock table contains at least one timed event. Refer to *The Clock Table* in Chapter 24 to learn how to schedule timed events.
Sequence	This LED lights when an autosampler sequence is running.
Non-Active Met/ Seq Edit	This LED lights when you press the EDIT/ACTIVE key to edit a method or sequence other than the one currently running. Press EDIT/ACTIVE again to return to the active mode.

The Oven Ramp LEDs

The oven ramp LEDs indicate the temperature ramp stages during a run. You can follow the progress of a run by observing these LEDs. Figure 2-3 shows the oven ramp LEDs.

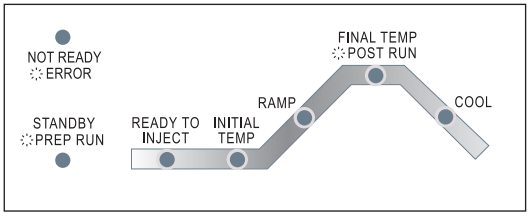


Figure 2-3. Oven Ramp LEDs

Table 2-2 describes the oven ramp LEDs.

Table 2-2. Oven Ramp LED Descriptions

LED	Description
Ready to Inject	This LED lights when the prep run has finished, indicating you can inject a sample or start an autosampler.
Initial Temp	This LED lights when a run starts and remains lit during the initial hold time.

Table 2-2. Oven Ramp LED Descriptions (Continued)

LED	Description
Ramp	This LED lights when the temperature starts to rise for the first ramp and remains lit until the last ramp's temperature has been reached.
Final Temp/Post Run	This LED lights during the final temperature holding time of last ramp and blinks during post-run procedures.
Cool	This LED lights while the oven returns to initial conditions.

The TRACE GC Ultra Keypad

The following sections list and describe the keys on the TRACE GC Ultra keypad. These keys are used to set up, operate, monitor, and program the instrument. Figure 2-4 illustrates the complete keypad.

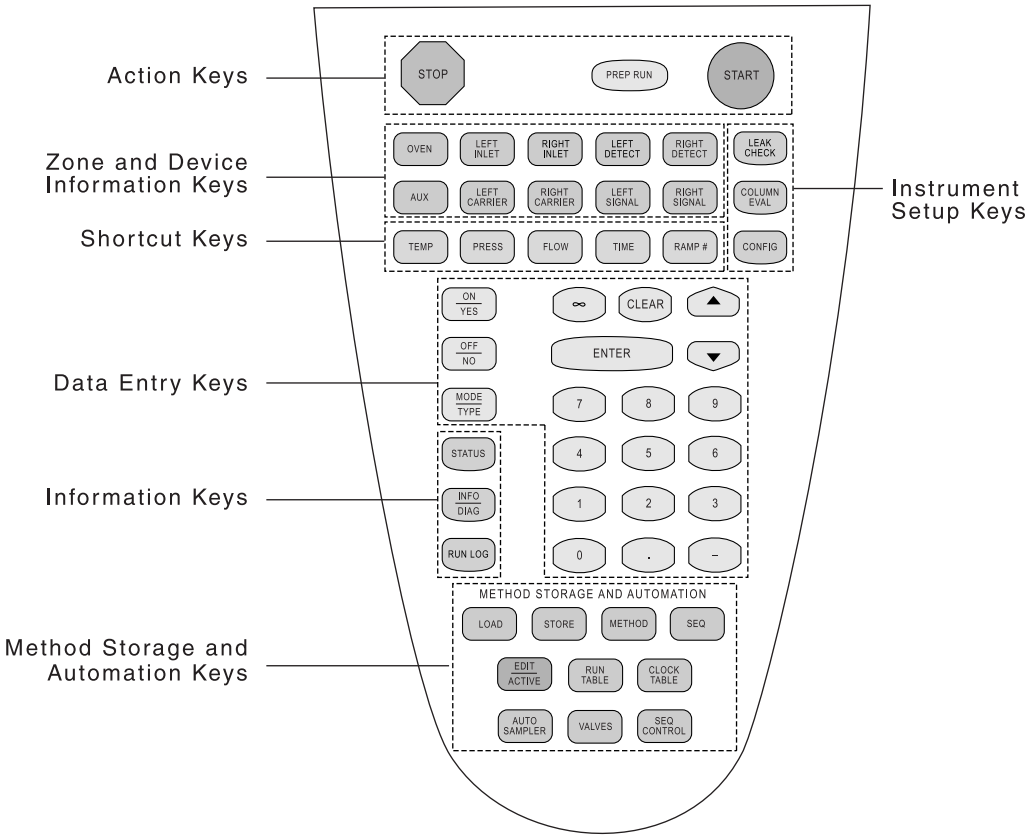


Figure 2-4. The TRACE GC Ultra Keypad

Action Keys

Use the three action keys to start or interrupt activities you have specified. For example, you can stop a run that you initiated.

The action keys are shown at the top of Figure 2-4 and in Figure 2-5.

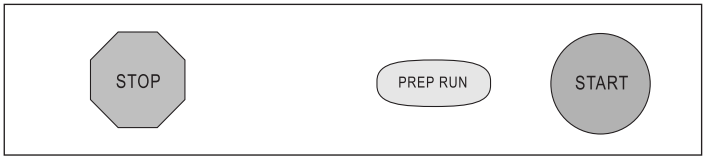


Figure 2-5. Action Keys

Start

The blue **START** key starts a run with programmed parameters after you manually inject a sample into an inlet. When a remote start by another device, such as an autosampler, has been programmed, the system automatically starts after injection.

CAUTION Do not inject a sample until the Ready to Inject LED is lit.

Stop

The red octagonal **STOP** key has the following functions:

- stops a run in progress
- resets the TRACE GC Ultra from **Ready** to **Not Ready**
- stops column characterization
- stops the leak check function

Prep Run

The light blue **PREP RUN** key activates operator-specified actions which must occur before the GC returns to **Ready to Inject** status. Press this key to return the TRACE GC Ultra to initial **Ready to Inject** status conditions for a run. This key activates septum purge conditions, prepares the injector for the type of injection you plan to use (split/splitless, etc.), and resets any gas saver features you have specified in the **LEFT** and **RIGHT CARRIER** menus. During a prep run, valves

open and close as necessary to prepare the injector before you make your injection. If `Ready Delay` is configured, this additional waiting time for external devices occurs after all other preparations are complete. The Standby/Prep Run LED stops blinking and stays lit to let you know when the GC can be moved to the **Ready to Inject** stage.

Zone and Device Information Keys

These brown keys open the zone and device menus. You enter setpoint for the GC column oven, injectors, detectors, carrier gases, and signals using these menus. Figure 2-6 shows the zone and device information keys.

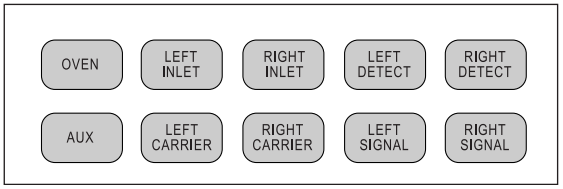


Figure 2-6. Zone and Device Information Keys

Oven

Use the **OVEN** key to set temperatures, times, and ramp rates. You can program up to seven temperature ramps per run. You can also program a timed postrun temperature.

When Ultra Fast Module device is installed, you can program up to three temperature ramps per run. For details refer to the UFM Ultra Fast Module Instruction Manual.

The cryogenic, or subambient, option allows you to specify oven temperatures lower than room temperature.

Left Inlet/Right Inlet

The parameters displayed when you press the **LEFT INLET** or **RIGHT INLET** key vary depending on the type of inlets installed in your TRACE GC Ultra system. Use these keys to set inlet parameters such as pressure and temperature and to turn the pressure and temperature on or off. Any pressure surge information you specify in the **`LEFT`** and **`RIGHT INLET`** menus will override other specified

gas pressure information. See the inlet chapters in Section III for more information.

Left Detector/Right Detector

The items displayed in the detector menus depend on the type of detectors installed and configured on your GC. The TRACE GC Ultra supports seven detectors:

- FID (Flame Ionization Detector)
- NPD (Nitrogen Phosphorus Detector)
- ECD (Electron Capture Detector)
- FPD (Flame Photometric Detector)
- PID (Photoionization Detector)
- TCD (Thermal Conductivity Detector)
- PDD (Pulsed Discharge Detector)

Any two may be installed at one time. If you have purchased the auxiliary detector option, you may install an ECD detector in tandem with one of the others.

Aux

This option controls external devices used with the TRACE GC Ultra. It is most commonly used for a stacked detector, Dual FPD, third detector base body, jet separator, valve oven, and MS (mass spectrometer) transfer line.

Left Carrier/Right Carrier

The items displayed in the carrier menus vary with the pressure and flow modes you select.

You have a choice of four flow modes:

- constant pressure mode, which sets pressure only

- constant flow mode, the most often used mode, which maintains a specific flow rate through the column

NOTE

In constant pressure mode, you can set pressure but not flow. In constant flow mode, you can set flow but not pressure.

- programmed pressure, which allows you to program up to three ramps of pressure changes
- programmed flow, which allows you to set a certain flow rate and increase it with up to three ramps

Refer to Chapter 4, *Digital Gas Control*, for more information about carrier menu options.

LEFT SIGNAL
RIGHT SIGNAL

Left Signal/Right Signal

The items displayed on the signal menus depend on the type of detector you have assigned to that location. Options for the ECD are somewhat different from the others.

The first item displays a unitless digital representation of the detector output. The other items help make that output more measurable and meaningful.

Instrument Setup Keys

Use the pink instrument setup keys to perform certain preparatory functions. Figure 2-7 shows these keys.

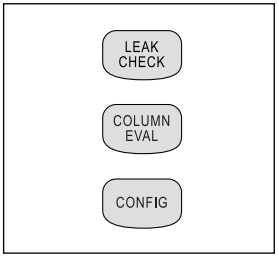


Figure 2-7. Instrument Setup Keys

COLUMN EVAL

Column Evaluation

This function allows you to calibrate the DCC module with the nominal column dimensions or, more accurately, according to the actual flow of the carrier gas measured at the outlet of the column.

NOTE If you use packed columns, you do not need to perform column evaluation.

LEAK CHECK

Leak Check

Press this key to perform a leak check at the desired pressure.

CONFIG

Config

Use this function to configure your TRACE GC Ultra hardware when you first receive it or make changes to it, such as when you install a new detector. See Chapter 3, *Configuration*, for more information.

Shortcut Keys

The light-brown shortcut keys display status of several GC parameters and allow you to jump within menus or to another menu to make adjustments. Figure 2-8 shows the shortcut keys.

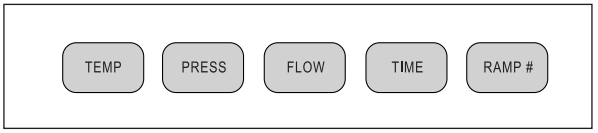


Figure 2-8. Shortcut Keys

TEMP

Temp

Press the **TEMP** key to display the setpoint and actual temperatures in menus with multiple parameters. You can also jump between multiple temperature parameters in menus, such as the **OVEN** and **LEFT** or **RIGHT INLET (PTV)** menus.

PRESS

Press

Use the **PRESS** key to display the setpoint and actual pressure readings and to go to the relevant fields in the carrier and inlet menus.

Flow

Press the **FLOW** key to display actual and setpoint gas flows for the inlets, columns, and detectors. You can jump to flow parameter fields in the inlet, carrier, and detector menus.

Time

Press the **TIME** key to display:

- time
- date
- last run time
- next run time
- elapsed time and time remaining during the current run
- the flow calculator

Ramp

Press the **RAMP #** key and a number to quickly edit a specific temperature or flow or pressure ramp.

Data Entry Keys

Use the light blue data entry keys shown in Figure 2-9 to enter information in the various menus.

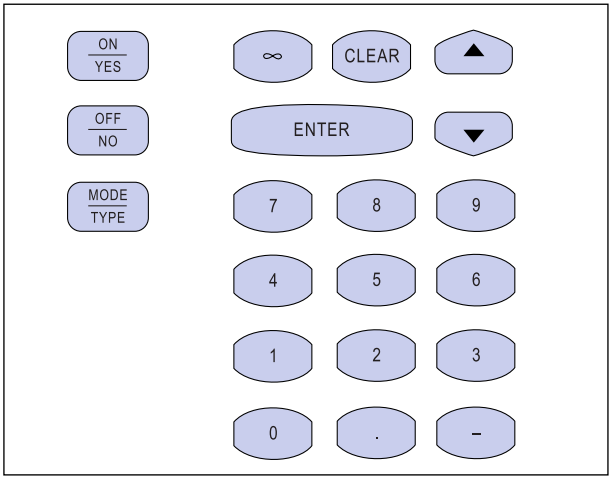


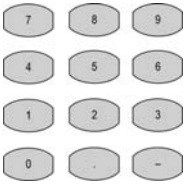
Figure 2-9. Data Entry Keys

On/Yes, Off/No

Use these keys to turn functions on or off and to answer yes or no questions.

Mode/Type

Use this key to display submenus for menu items that do not have yes/no or on/off choices. Usually you can use the **ENTER** key for the same purpose.



Numeric

The numeric keypad includes numbers from 0–9. The keypad includes a decimal point, minus key, and infinity. The minus key acts as a negative sign (for entering subambient temperatures) and a range key (for entering sets of numbers such as 1–30).

∞

Use this key to enter infinite times or durations.

Enter

You can use this key to:

- confirm changes to a selected menu item. For instance, after you have selected `Off` as the status for a function, press **ENTER**.
- confirm typed information in memory. For instance, after you have typed `250` as your setpoint oven temperature, press **ENTER**.
- move to a submenu. For instance, press **ENTER** when you have selected `Detector: FID-A` to move from the **`CONFIG RIGHT DETECTOR`** menu to the **`DETECTOR TYPE`** menu. You can use the **MODE/TYPE** key for the same purpose.
- start or stop the timer on the stopwatch feature.

Clear

You can use this key to:

- clear a field in which you have started to enter data.
- back up to the previous menu level. For example, after you have chosen a detector type from the **`DETECTOR TYPE`** menu, press **CLEAR** to return to the **`CONFIG RIGHT DETECTOR`** menu.
- remove programmed events such as:
  - clock time events
  - run time events
- clear a nonfatal error message and return to the previous display.
- reset the timer in the stopwatch feature.

Arrows

Use the arrow keys to scroll through a list of menus or to move the cursor to an editable field.

Information Keys

The pink information keys shown in Figure 2-10 provide status, menu, diagnostic, and run error data.

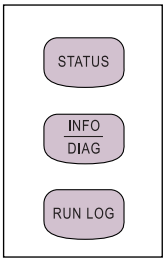


Figure 2-10. Information Keys

Status

This function displays the instrument status and any reasons the GC is in **Not Ready** mode.

Info/Diag

Press this key once to bring up the range, options, and function of a selected menu item, if the item can be edited.

Press the key twice to bring up diagnostic information, including:

- software and hardware information
- power checks
- oven, injector, and detector status

Run Log

This function displays the run log, which records errors that happen during run time. It displays the time and description of any deviations that occur. See *Run Log* in Chapter 24 on page 461 to learn how to use this feature.

Method Storage and Automation Keys

A *method* controls the function of the gas chromatograph during analytical runs. You may specify parameters for any zone and device (including temperature ramps in the oven menu), as well as autosampler parameters and run table timed events. See Chapter 26, *Using Analytical Methods*, for more information.

A *sequence* describes how samples are treated in the injection stage and what method will be used to analyze them.
For information about sequences, refer to:

- Chapter 27 *AI 3000 / AS 3000 Autosampler Sequences*

Use the brown keys and the blue **EDIT/ACTIVE** key shown in Figure 2-11 to automate and edit certain functions.

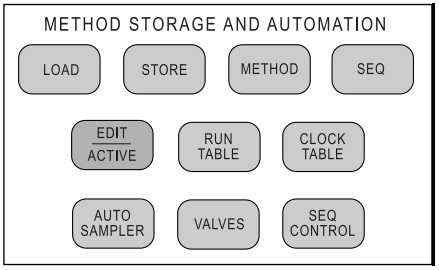


Figure 2-11. Method Storage and Automation Keys

Load

If you don't have a data system, use this feature to recall an analytical method or autosampler sequence. For instance, pressing LOAD and choosing `Sequence` and then `5` from subsequent menus will bring up the parameters of sequence #5.

Store

Use this feature to enter an analytical method and/or autosampler sequence into memory.

Method

Use this feature to load, store, or edit an analytical method with programmed temperature and pressure ramps. You can store 10 methods in the TRACE GC Ultra in addition to the default method.

Seq

Use this feature to load, store, or edit a handling sequence for samples in an autosampler tray. You can store five sequences with five subsequences each.

Edit/Active

Press the EDIT/ACTIVE key to edit an inactive sequence or method while another is running. Your changes do not affect the current run. Press EDIT/ACTIVE again to leave the editing mode and to display menus for current run.
To learn how to develop methods and sequences refer to:

- Chapter 26, *Using Analytical Methods*, or
- Chapter 27 *AI 3000 / AS 3000 Autosampler Sequences*

Run Table

Use this feature to program events to occur during a run, such as a valve opening. You can specify up to ten events for each of ten methods.

Clock Table

Use this feature to program up to ten events to occur in real time. For instance, you could specify a column bakeout on Wednesday at 8:00 A.M. These events cannot be stored in a method. When the `Mode:` parameter is set to `Active`, you can program the days as a `Specific cycle`.

Auto Sampler

Use this feature to control all autosampler functions except alignment. From the TRACE GC Ultra or the data system you can specify prewash and postwash instructions, injection methods, and number of injections per vial.

If you don't have an autosampler, you will receive an error message when you press this key or any key or menu item associated with the autosampler or its sequences (**SEQ**,

SEQ CONTROL, **LOAD»**`Sequence`, **STORE»**`Sequence`, or **EDIT/ACTIVE»**`Sequence`). Press any key to return to your previous menu.

Valves

Use this feature to specify and control up to four valves, as well as to manually change the state of inlet valves or oven valves.

Seq Control

Use this feature to start or interrupt a sequence.

General Navigation

The display shows three menu items at a time. A menu title bar in capital letters always appears. Use the **ARROW** keys to scroll through the menus. To see a submenu, press **ENTER** or **MODE/TYPE**. To return to a higher-level menu, press **CLEAR**. Table 2-3 displays examples of the various types of menu items and the ways to edit their fields.

Table 2-3. Sample Menu

Menu	Editing Instructions
LEFT INLET (S/SL) ↕	The title bar is always displayed. It cannot be edited. It can change, though, depending on your choices in other menus. For example, if you select an S/SL inlet, this title changes to **LEFT INLET (S/SL)**. The arrow indicates that more items are available than the ones appearing in the four-line display.
Mode: splitless	Press **ENTER** or **MODE/TYPE** to display the submenu for this item.
Total flow (57.0)	The parentheses indicate that this field cannot be edited. Use the arrow keys to scroll to another menu item.
Split flow 50 50	The number on the left is the actual value. The number on the right is the setpoint. Use the numeric keypad to enter an integer to change the setpoint, or press the **OFF/NO** key to turn off the option.
Splitless time 1.00*	This line shows a time entry. Use the numeric keypad to enter a number with up to two decimal places. The asterisk shows that it is being edited. An asterisk can also indicate the active selection in a list.
Const sep purge? Y<	Use the **ON/YES** and **OFF/NO** keys to edit this item. The arrow shows that this item is selected.

OPERATING SEQUENCE

Editing a Menu Item

1. Press the relevant key to select the menu to be edited, for instance, OVEN.
2. To select an item within the menu, use the arrow keys to scroll until the cursor (<) points to the item you want to edit.
3. You can change the field's content in several ways:
   a. To choose `On/Off` or `Yes/No`, use the **ON/YES** and **OFF/NO** keys.
   b. Enter a number with the numeric keypad.

NOTE You cannot edit any item in parentheses.

   c. If the field cannot be filled with on/off, yes/no, or a number, press **ENTER** or **MODE/TYPE** to display a submenu of choices. In the submenu, you may use the keypad or scroll with the arrow keys.

NOTE Press the **INFO/DIAG** key once to display the selected field's range and options. If the field cannot be edited, no information will appear. Press **CLEAR** to return to the menu.

4. When you have entered the proper information in the field, press **ENTER** to load the new setpoint. The blinking asterisk disappears after you press **ENTER**. To erase an entry before choosing it, press **CLEAR**.

NOTE If you are working in a submenu, you can also use the **CLEAR** key to return to the higher-level menu.

5. Use the **ARROW** keys to scroll to the next item you want to edit.

Error Conditions

When error conditions occur, a message will appear on the display and the Not Ready/Error LED will blink. For minor conditions such as trying to specify parameters for items that haven't been installed, the TRACE GC Ultra will display a `Not installed or not configured` message.

However, the TRACE GC Ultra shuts down under some error conditions, such as unbounded gas flow, hydrogen leaks, and improperly installed or configured heating devices. When the TRACE GC Ultra detects these potential hazard conditions, it shuts down.

Unbounded Gas Flow

The TRACE GC Ultra shuts down when it senses unbounded gas flow. You need to repair the source of the gas flooding and restart the instrument.

Hydrogen Leak

You can choose hydrogen as a carrier gas only if a hydrogen sensor was installed at the factory. Refer to *Using the Hydrogen Sensor* on page xxix for information on this option.

If the hydrogen sensor detects a leak, the TRACE GC Ultra shuts down. You need to find and repair the leak before restarting.

Thermal Shutdown

The TRACE GC Ultra hardware and software protect the system from *thermal runaways* or uncontrolled temperatures. The hardware and software check for different thermal error conditions and shut down the heated zones if any errors are detected.

For proper operation, all potential heated zones must have either an **installed sensor**, or a **jumper**.

If a jumper is used, the heated zone must not be configured.

Hardware Shutdown

The temperature sensors in the TRACE GC Ultra create a closed circuit. If a sensor fails, the circuit opens and the hardware initiates thermal shutdown. This shutdown can also happen if hardware containing a temperature sensor, such as an ECD, is removed. The missing temperature sensor opens the circuit and the hardware initiates thermal shutdown. You must connect a plug to the temperature sensor cable to close the circuit when an ECD is removed from the system.

When the hardware initiates a thermal shutdown, the TRACE GC Ultra will display the following message:

```
 TEMPERATURE SHUTDOWN

Temperature zone(s)
exceeded the allowed
hardware limit(s)
```

If a hardware thermal shutdown occurs because of a failed temperature sensor, contact your local Thermo Fisher Scientific customer service representative for assistance. Refer to Appendix B, *Customer Communication*, for a list of Thermo Fisher Scientific offices and affiliates worldwide.

Software Shutdown

The TRACE GC Ultra software uses the temperature sensors to control the temperature zones. If the software is unable to control the temperature because the leads to the temperature sensor are crossed, the software will initiate a thermal shutdown. If the system is configured for hardware containing a temperature sensor (such as an ECD), the software will initiate thermal shutdown if that hardware is removed.

When the software initiates a thermal shutdown, the TRACE GC Ultra displays one of the following messages:

```
 TEMPERATURE SHUTDOWN

Isothermal zone not
controlling or
heating
```

```
 TEMPERATURE SHUTDOWN

Shorted temp sensor
Run temperature
diagnostics
```

If a software thermal shutdown occurs, do the following:

1. Configure the instrument properly, following the configuration instructions in Chapter 3, *Configuration* and in Chapter 15, *Detector Overview*.

2. Shut down the TRACE GC Ultra and turn it on again.

3. Resume operating your TRACE GC Ultra.

3

Configuration

This chapter describes how to set up the software on your TRACE GC Ultra either to match the installed hardware or to reflect your preferences.

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Left/Right Detector 76
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When to Configure

The TRACE GC Ultra has few special set up sequences. After you first install and configure the instrument, you will need to reconfigure only after you make changes to the components. You must configure the system when:

- using the TRACE GC Ultra for the first time
- adding new components
- changing detectors
- changing carrier gases
- changing column types (to set the appropriate maximum oven temperature)
- replacing a detector board
- changing to an analytical method that requires different hardware

Configuration Main Menu

Press **CONFIG** to open the **CONFIGURE** main menu. The menu items may change, depending on factory settings and current hardware. For instance, if no right inlet is installed, the right inlet item will not appear in the **CONFIGURE** menu. If you press the **RIGHT INLET** key, the following message displays:

```
     RIGHT INLET

Not present, or not
configured
```

Table 3-1 describes the items in the **CONFIGURE** menu. Each item has a submenu.

Table 3-1. Configuration Main Menu

Menu	See…	Comments
CONFIG		This line is the menu title bar.
Oven	page 72	Controls preparatory actions such as automatic prep run, timeout, equilibration time, and ready delay time. It also enables cryogenic options and specifies maximum oven temperature.
Active inlet		This parameter indicates which inlet is operating when a three-way valve is installed.
Left inlet	page 75	This parameter controls the mode for the left inlet.
Left carrier	page 76	This parameter controls the type of carrier gas for the left inlet.
Right inlet	page 75	This parameter controls the mode for the right inlet.
Right carrier	page 76	This parameter controls the type of carrier gas for the right inlet.
Left detector	page 76	This parameter controls the type of detector fuel gas and make-up gas for the left detector.
Right detector	page 76	This parameter controls the type of detector fuel gas and make-up gas for the right detector.
Aux detector		This parameter controls the type of detector fuel gas and make-up gas for the auxiliary detector.
Aux. Zones	page 78	This parameter controls the temperature for preconfigured devices. Two auxiliary temperature zones are available.
Time	page 78	This parameter sets the time and date.
Valves	page 80	This parameter configures up to eight external valves sampling and/or switching valves when present.
Autosampler	page 80	This parameter controls an autosampler.
Handshaking	page 81	This parameter configures the polarity of signals from external devices.
Keyboard & display	page 83	This parameter controls keyboard and display preferences.

Oven

The TRACE GC Ultra oven provides great flexibility in controlling and programming temperatures. In the **CONFIG OVEN** menu of Table 3-2, you can set various preparatory parameters as well as activate the cryogenic option, if your GC has that equipment.

When Ultra Fast Module device is installed, a dedicated **CONFIG OVEN** menu will be visualized as reported in the *UFM Ultra Fast Module Instruction Manual*.

Table 3-2. Config Oven Menu

Parameter	Range/Options	Comments
CONFIG OVEN		This line is the menu title bar.
Auto prep run	On/Off	This parameter automatically performs run preparations when a sequence is active.
Auto Start	On/Off	Allows an automatic *Start* signal.
PR timeout	0.00–99.9 min	This parameter returns the GC to standby mode if injection does not occur by the time set and Auto prep run is set to Off.
Enable cryogenic?[1]	Yes/No	This function enables the oven's cryogenic system when it is installed and configured. Press YES to activate the cryogenic system. Press NO to deactivate it.
Cryo Timeout[1]	0.00–999.99 min	This parameter specify the time at which the cryo system will be disabled if during the cooling phase the GC does not reach the initial temperature.
Start cryo at[1]	40 to 200 °C	This parameter specifies the temperature at which the cryo system begins to supply the coolant.
Equil time	0.00–999.99 min	This parameter allows the oven temperature to stabilize after cooling for the length of time set
Ready delay	0.0–99.9 min	This parameter allows additional waiting time after the GC is ready to ensure that any external devices are also ready.
Max temp	0–450 °C	This parameter limits the oven temperature to the setpoint.

1. These items appear only when cryogenic equipment has been installed and configured at the factory.

Left/Right Inlet

The **LEFT** and **RIGHT INLET** menus allow you to configure the type of column inlet you will be using. The settings for split/splitless, packed column, purged packed column, and programmable temperature vaporizing injectors have been preset at the factory, but you may select from the three types of on-column injectors. Table 3-3 displays the **CONFIG RIGHT INLET** menu and its submenu when an on-column inlet is installed.

Table 3-3. On Column Inlet Configuration Menu

Menu	Submenu	Comments
CONFIG RIGHT INLET Inlet type OCI <	RIGHT INLET OCI < OCHOT LVOCI	Press **ENTER** or **MODE/TYPE** to enter the submenu. Scroll with the **ARROW** key until the cursor points to your selection. Press **ENTER** to choose it. Press CLEAR to return to the main **CONFIGURE** menu.

NOTE To run the OCHOT option, the TRACE GC Ultra needs a special temperature sensor. The sensor is installed and configured at the factory.

If you select LVOCI and your instrument does not have a solvent vapor exit valve, the TRACE GC Ultra will shut down and display the temperature fault message shown in Figure 3-1. Select another option and restart the instrument.

```
TEMPERATURE SHUTDOWN

Shorted temp sensor
Run temperature
diagnostics
```

Figure 3-1. Thermal Shutdown Message

Left/Right Carrier

The left and right carrier menus let you select a carrier gas for each column.

Table 3-4. Left/Right Carrier Menu

Menu	Gas
RIGHT CARRIER	This line is the menu title bar.
He	This selects helium.
H2[1]	This selects hydrogen.
N2	This selects nitrogen.
Ar/CH4 5%	This selects argon/5% methane.
Ar	This selects argon.

1. You cannot choose hydrogen as a carrier gas unless your instrument has a hydrogen sensor. See *Using the Hydrogen Sensor* on page xxix for more information.

Left/Right Detector

The TRACE GC Ultra works with seven types of detectors:

- FID (Flame Ionization Detector)
- NPD (Nitrogen Phosphorus Detector)
- ECD (Electron Capture Detector)
- PID (Photoionization Detector)
- FPD (Flame Photometric Detector)
- TCD (Thermal Conductivity Detector)
- PDD (Pulsed Discharge Detector)

Because the TRACE GC Ultra has three detector board slots, you may alternate between your choice of three detectors. When you purchased your TRACE GC Ultra system, you specified which detectors and what options you required. Some of the configuration was done at the factory, but you can assign a column to a specific detector.

To change a detector, you must do the following:

- mount and connect the detector
- configure the GC and the data system
- plumb the appropriate gas supplies as described in Chapter 15, *Detector Overview*

The items in the **RIGHT** and **LEFT DETECTOR** menus vary, depending on the detectors installed. To see the available detectors, scroll to `Right Detector` or `Left Detector` in the **CONFIGURE** menu, then press **MODE/TYPE** or **ENTER**. A list of detectors and their board locations appears:

```
DETECTOR TYPES
*  FID-A
   NPD-B
   ECD-C
```

The letters `A`, `B`, and `C` next to the detector refer to the three available board slots in the TRACE GC Ultra. You can assign any of the available detectors as either the right or left detector.

Example: Selecting an FID for the Left Detector

1. Press **CONFIG** and scroll to `Left Detector`.
2. If your GC has the same options as those shown above, your menu selections will be `FID`, `NPD`, and `ECD`. Scroll to `FID` and press **ENTER**.

If you want to change detectors and all detectors have been assigned, you must choose one port and first set it to `none` before you can choose another detector.

If you want to change to a detector with a secondary heating element (ECD), exchange the detector before changing the configuration. Changing the configuration first could cause a thermal shutdown.

Example: Changing the Right Detector from an FID to an NPD

The types of detectors supported by DGFC depend on the installed detector modules. Refer to Table 15-2 in Chapter 15, *Detector Overview*, for the correct gas supply connections to the detector module inputs.

1. Press **CONFIG**, then press **RIGHT DETECT**.
2. Select `NPD` and press **ENTER**.

Each kind of detector has its own settings and parameters.

NOTE

The NPD requires an *AD* type DGFC control module. This module can be used to control FID flame gases. To do this, you must plumb the hydrogen supply to the Gas 1 input of the DGFC module and leave the Gas 3 input unconnected.

Auxiliary Zones

The two auxiliary temperature zones control temperature in extra hardware such as a heater, temperature sensor, valve oven, jet separator, mass spectrometer interface, or other end-user devices. These resources must be configured at the factory. However, in this menu you can specify whether to heat the assigned zone. Table 3-5 illustrates the **`Auxiliary Temp Zone`** menu.

Table 3-5. Auxiliary Zone Options

Menu	Options	Comments
`AUXILIARY TEMP ZONE`		This line is the menu title bar.
`Aux 1 zone active N <`	Yes/No	These parameters control the temperature for preconfigured devices
`Aux 2 zone active N`	Yes/No	

Time

You can set events, such as a column bakeout, to happen at certain times of the day on certain days. Refer to Chapter 24, *Automated Functions*, for more information about programming clock time events.

The clock time events refer to the time set in the TRACE GC Ultra's clock. You can set this time from the **CONFIGURE** menu.

When you open the **CONFIG TIME** menu, the following items appear:

- `Time (hhmm)`
- `Date (mmddyy)`

Time is set on a 24-hour clock.

OPERATING SEQUENCE

Setting the Time

1. Press CONFIG to open the **CONFIGURE** menu.
2. Scroll to `Time` and press **ENTER**.
3. Scroll to `Time (hhmm)`.
4. Use the numeric keypad to enter the time. For example, for 8:05 A.M., type `0805`. For 2:30 P.M., type `1430`.
5. Press **ENTER**. Press **CLEAR** to return to the **CONFIGURE** menu.

OPERATING SEQUENCE

Setting the Date

1. Press CONFIG to open the **CONFIGURE** menu.
2. Scroll to `Time` and press **ENTER**.
3. Scroll to `Date (mmddyyyy)`.

4. Use the numeric keypad to enter the month, day, and year. For example, for September 7, 2009, type `09072009`.

5. Press **ENTER**. Press **CLEAR** to return to the **CONFIGURE** menu.

Once you set the time and date, the values are battery backed-up and will remain even after you turn off the instrument.

Valves

Press CONFIG to open the **CONFIGURE** menu, then scroll to `Valves` and press ENTER to open the **CONFIGURE VALVES** menu where you may configure up to eight external valves.

```
CONFIGURE VALVES
 Valve#1        <
 ---------------
 Valve#8
```

From each line press ENTER to open the menu where you may configure the type of valve.

```
CONFIGURE VALVE#1
 *Gas Sampling
  Switching
  None
```

Select the valve type of your interest and press ENTER.

To program these valves refer to Chapter 12 and Chapter 25.

Autosamplers

Most autosampler functions can be controlled from the TRACE GC Ultra or the data system. Only the alignment must be programmed at the autosampler.

Press CONFIG to open the **CONFIGURE** menu, then scroll to `Autosampler` and press ENTER to open the **CONFIG AUTOSAMPLER** menu.
Table 3-6 describes an example of autosampler configuration options.

Table 3-6. Autosampler Configuration

Menu	Range	Comments
CONFIG AUTOSAMPLER		This line is the menu title bar.
Program inj speed	Yes/No	This parameter allows you to specify a slower plunger speed, such as for a large volume injection.
Use internal standard	Yes/No	This parameter allows you to inject an internal standard with the sample.

Handshaking

The TRACE GC Ultra can cooperate with other instruments, such as an autosampler or mass spectrometer, during analysis.
To allow other devices to run properly, you must indicate how the signal will change.
For example, the menu in Table 3-7 specifies that another device will start the GC when the remote start signal changes from high to low.

Press CONFIG to open the **CONFIGURE** menu, then scroll to `Handshaking` and press ENTER to open the **HANDSHAKING** menu.

Table 3-7. Handshaking Configuration Menus

Menu	Submenus	Comments
HANDSHAKING		This line is the menu title bar.
Remote start in	REMOTE START Input pulse: Low to High High to Low	This parameter allows another device to start the GC. For the AI 3000/AS 3000 autosampler, you must select `High to Low`.
Inhibit READY in	INHIBIT READY Inhibit readiness: When high When low	This parameter delays readiness until the GC receives a signal from another device.
End of run out	END OF RUN Output pulsed: Low to High High to Low	This parameter signals another device, such as an integrator, that the run has ended.
Start of run out	START OF RUN Output pulsed: Low to High High to Low	This parameter signals another device, such as an integrator, that the run has started.
GC READY out	READY OUT Show readiness: When high When low	This parameter signals another device that the GC is ready.
Prep Run out	IN PREP RUN Indicate preparing: When high When low	This parameter signals another device that the GC is preparing for a run.

Keyboard and Display

This menu allows you to customize your keyboard and display. Table 3-8 describes these options.

`Keyboard beep` leads to a submenu where you can specify when you want the GC to alert you with a keyboard sound. To move to the submenu, select `Keyboard beep` and press **ENTER** or **MODE/TYPE**.

Table 3-8. Keyboard & Display Menu

Menu	Options	Comments
KEYBOARD & DISPLAY		This line is the menu title bar.
Keyboard lock <	On/Off	This parameter prohibits any menu edits.
Keypad beep	Refer to Table 3-9.	This parameter causes the GC to beep when you press the keys specified in the submenu.
Warning beep	On/Off	This parameter causes the GC to beep for certain error conditions, such as low carrier gas pressure or unbounded flow.
Delimiter type	. or,	This option allows you to select a period or comma as a decimal marker.
Pressure units	kPa, psi, bar	This option allows you to select the pressure unit for display.
Run log active	Yes/No	This option activates a run log during a run.

Table 3-9 describes each of the keyboard beep options.

Table 3-9. Keyboard Beep Submenu

Menu	Options	Comments
KEYBOARD BEEP		This line is the menu title bar.
Any key press	On/Off	This parameter causes the GC to beep when you press any key on keypad.
Enter key press	On/Off	This parameter causes the GC to beep when you press **ENTER**.
On invalid key	On/Off	This parameter causes the GC to beep when the key you press is not a valid option, such as a numeric entry instead of **ON**/**OFF**.
Never	On/Off	This option turns off keyboard beeps.

SECTION

II

Gases Control

This section contains information on controlling and programming the detector and carrier gas flows to the TRACE GC Ultra.

Chapter 4, *Digital Gas Control*, describes the automatic (DCC and DGFC) gas control features of the TRACE GC Ultra and contains the instructions to program and regulate the GC carrier gases control.

4

Digital Gas Control

This chapter describes the automatic DCC and DGFG gas control features of the TRACE GC Ultra and contains the instructions to program and regulate the GC carrier, detector and auxiliary gases control.

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DCC Carrier Gas Control 90
DGFC Detector Gases Control 92
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Gas Control with DCC and DGFC Modules

The GC electronically control all the gas flows and pressures in the instrument. It provides:

- flow and/or pressure control for all injectors, including flow and pressure programming for carrier gas
- flow control for all detector gases
- a gas saver mode to reduce carrier gas consumption with the Split/Splitless (S/SL) and the Programmable Temperature Vaporizing (PTV) injectors.

The GC automatically identifies injectors and detectors with electronic control modules during power-up. Some information must be entered manually by the user. This operation is called *configuration*.

Gas Supplies

The configuration of your TRACE GC Ultra determines the carrier, make-up, and fuel gas requirements. The gas flow modules installed determine whether you regulate the gas flow and pressure through digital DCC and DGFC pneumatic control.

You should not connect any gases to the TRACE GC Ultra that are not referenced in the documentation.

Commonly used gases are nitrogen, helium, hydrogen, and air. Other gases such as argon and argon/methane are used more rarely. The gases required for different injectors and detectors are discussed in detail in Chapter 1 of the TRACE GC Ultra *Site Preparation and Installation Manual*.

Pressure Units

You can specify the pressure units the TRACE GC Ultra displays. The default pressure unit is the kilopascal (kPa). You specify the pressure units in the **CONFIGURE** menu as described in the *Configuring the Pressure Unit* operating

sequence on page 89. Table 4-1 gives a brief conversion guide for the most commonly used pressure units in gas chromatography.

Table 4-1. Pressure Units Conversion

To convert	To	Multiply by
kPa	bar	0.01
	psi	0.145
bar	kPa	100
	psi	14.51
psi	kPa	6.89476
	bar	0.0689476

100 kPa = 1 bar = 14.51 psi

OPERATING SEQUENCE

Configuring the Pressure Unit

The pressure unit is already configured to kPa (kilopascals). To change the configuration, proceed as follows:

1. Press CONFIG, scroll to `Keyboard and Display`, then press ENTER.
2. Scroll to `Pressure Unit` and press ENTER to open the **`PRESSURE UNITS`** menu.

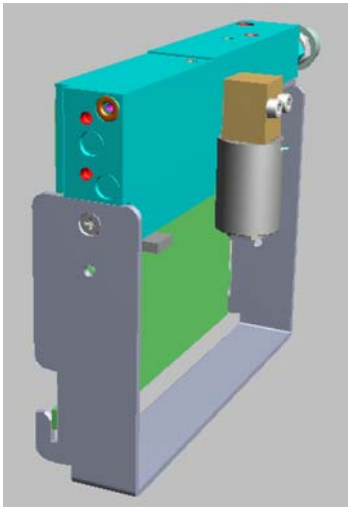
```
        PRESSURE UNITS

  psi
* kPa                        <
  bar
```

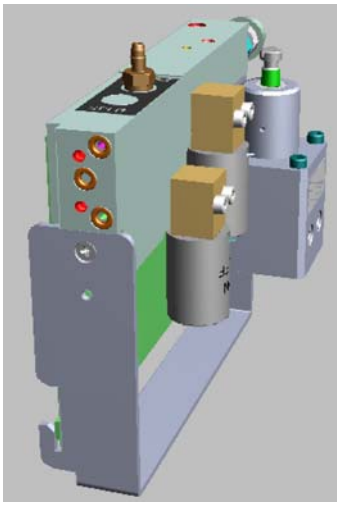
3. Scroll to the pressure unit to be used and press ENTER twice to confirm the selection. An asterisk appears to the left of the pressure unit selected.

DCC Carrier Gas Control

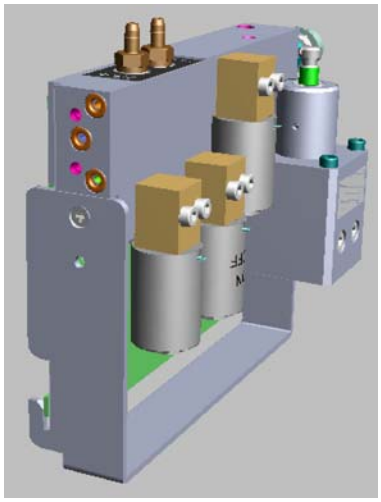
There are three types of DCC modules as shown in Figure 4-1. The type of module installed depends on the injector in use. Each type of DCC module is available into two versions according to the full scale (f.s.) of the flow regulator.



1. DCC Module (250 kPa or 1000 kPa f.s.) for OCI and PKD Injectors



2. DCC Module (250 kPa or 1000 kPa f.s.) for PPKD Injector



3. DCC Module (250 kPa or 1000 kPa f.s.) for S/SL and PTV Injectors

Figure 4-1. DCC Modules

You enter the DCC gas control setpoints in the **CARRIER** and **INLET** menus.

The carrier gas menu includes all of the parameters for controlling gas flow. For a detailed description of the **CARRIER** menu items and ranges, refer to paragraph *Carrier Gas Menu* on page 93.

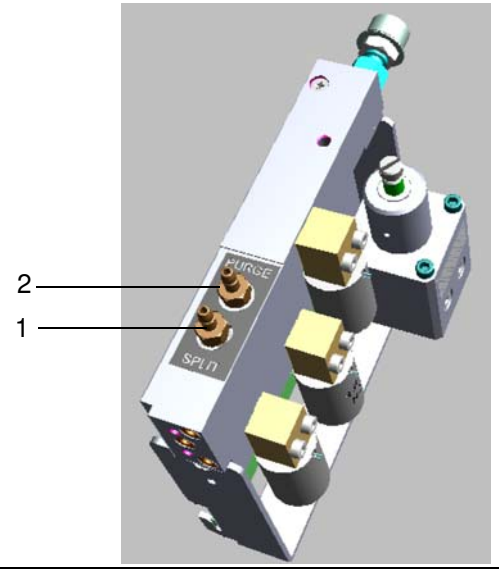
For a detailed description of the **INLET** menu, refer to the relevant chapter according to the injector in use.

The electronic control of the carrier gas allows also the following operations.

- **Column Evaluation**
  Refer to the *Performing a Column Evaluation* operating sequence in Chapter 14 for more information.
- **Leak Check**
  Refer to the *Performing a Leak Check* operating sequence in Chapter 14 for more information.

DCC Gas Flow Vents

When present, the septum purge and the split flow exit through the vents on the top of the instrument as shown in Figure 4-2.



1. Split Line Vent
2. Septum Purge Line Vent

Figure 4-2. DCC Split Flow and Septum Purge Flow Vents

DGFC Detector Gases Control

There are four types of DGFC modules as shown in Figure 4-3. The type of module installed depends on the detector in use and on the presence of the make-up gas line.

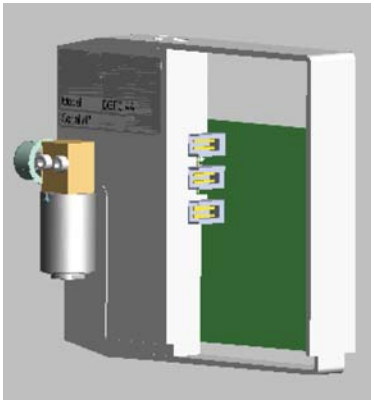
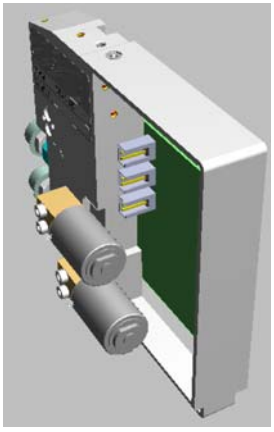
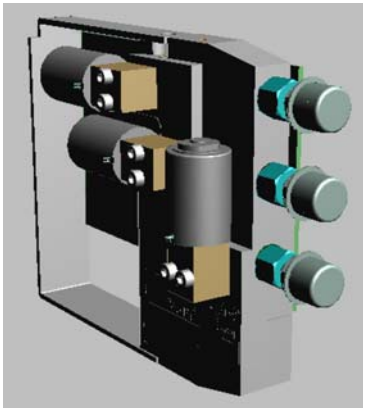
		
1. DGFC Module type AA for ECD only	2. DGFC Module type AB for ECD, PID, FPD, FID without Make-up gas	3a. DGFC Module type AC for ECD, PID, FPD, FID with Make-up gas 3b. DGFC Module type AD for NPD, ECD, PID, FPD, FID without Make-up gas

Figure 4-3. DGFC Modules

You enter the gas control setpoints in the **DETECTOR**, and **AUXILIARY** menus. Detector gases are discussed in Chapter 15.

Carrier Gas Menu

This paragraph explains the electronic programming and control of the GC carrier gas. The Digital Carrier Control (DCC) modules provide for the electronic control of the carrier gas.

The **CARRIER** menu includes the control parameters for the carrier gas, regardless of the carrier gas type.
Visualized parameters change according to the set operating mode: constant flow, constant pressure, programmed flow or programmed pressure.

Press LEFT CARRIER or RIGHT CARRIER to display the **LEFT** or **RIGHT CARRIER** menu.

```
        LEFT CARRIER[1]

 Pressure          30.0    30.0
 Col.flow          3.00
 Lin. veloc.             (60.9)<
```

1. These settings could also be for the right carrier.

Flow Mode

The `Flow mode` menu displays the four options available for the carrier gas control:

- constant flow
- constant pressure
- programmed flow
- programmed pressure

Scroll to the `Flow mode` parameter and press MODE/TYPE or ENTER to open the flow mode menu:

```
   LEFT CARRIER FLOW MODE
* Constant flow           <
  Constant pressure
  Programmed flow
  Programmed pressure
```

Scroll to the desired flow mode and press ENTER to confirm the selection. An asterisk appears to the left of the selected flow mode. The items in the **CARRIER** menu change depending on the selected flow mode. Tables 4-2 through 4-5 show the **CARRIER** menu for each of the four modes.

Constant Flow Mode

In constant flow mode, 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.

Table 4-2. Carrier Menu in Constant Flow Mode

Menu	Range	Comments
LEFT CARRIER		This line is the menu title bar.
Pressure	Not editable	This line displays the pressure setpoint value that depends on the flow set.
Col. flow	On/Off, 0–100 mL/min	This line shows the constant flow rate of carrier gas passing through the column. Press ON to display the actual and setpoint values. Press OFF or 0 to turn off all inlet flows.
Lin. veloc.	Not editable	This line shows the velocity of the carrier gas through the column, expressed in cm/s.
Void time	Not editable	This line indicates the elution time, expressed in seconds, of an un-retained peak.
Flow mode	Const flow	This line indicates the selected flow mode.

Table 4-2. Carrier Menu in Constant Flow Mode (Continued)

Menu	Range	Comments
Gas saver flow[1]	On/Off, 0–500 mL/min	This line indicates the gas saver flow. Press ON to turn on the gas saver flow and display the setpoint value. Press OFF to turn off the gas saver function. The flow is retained in memory.
Saver time[1]	0.00–999.99 min	This line shows the gas saver time, which is the time in the run at which the gas saver function starts to operate. This line does not appear if Gas saver flow is Off.
Vacuum comp	On/Off	Use this parameter only when the TRACE GC Ultra is used with a mass detector to compensate for vacuum column outlet.

1. This parameter is displayed only for the S/SL and PTV injector.

Constant Pressure Mode

In constant pressure mode, 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.

Table 4-3. Carrier Menu in Constant Pressure Mode

Menu	Range	Comments
LEFT CARRIER		This line is the menu title bar.
Pressure	On/Off, 2–250 kPa[1] or 10–1000 kPa	This line shows the constant pressure of the carrier gas passing through the column. Press ON to display the actual and setpoint values. Press OFF or 0 to turn off all inlet pressures.
Col. flow	Not editable	This line displays the actual column flow value that depends on the pressure set.
Lin. veloc.	Not editable	This line shows the velocity of the carrier gas through the column, expressed in cm/s.
Void time	Not editable	This line indicates the elution time, expressed in seconds, of an un-retained peak.
Flow Mode	Const Pres	This line indicates the selected flow mode.

Table 4-3. Carrier Menu in Constant Pressure Mode (Continued)

Menu	Range	Comments
`Gas Saver flow`[2]	On/Off, 0–500 mL/min	This line indicates the gas saver flow. Press ON to turn on the gas saver flow and display the setpoint value. Press OFF to turn off the gas saver flow. The flow is retained in memory.
`Gas Saver time`[2]	0.00–999.99 min	This line shows the gas saver time, which is the time in the run at which the gas saver function starts to operate. This line does not appear if `Gas saver flow` is `Off`.
`Vacuum comp`	On/Off	Use this parameter only when the TRACE GC Ultra is used with a mass detector to compensate for vacuum column outlet.

1. The default pressure unit is kPa. You can change the units to psi or bar in the **`CONFIGURE`** menu.
2. This parameter is displayed only for the S/SL and PTV injectors.

Programmed Flow Mode

In programmed flow mode, the column flow rate can be programmed to change during the analytical run. In this mode, up to three flow ramps can be entered.

Table 4-4. Carrier Menu in Programmed Flow Mode

Menu	Range	Comments
`LEFT CARRIER`		This line is the menu title bar.
`Pressure`	Not editable	This line displays the pressure value that depends on the flow set.
`Col. flow`	On/Off, 0–100 mL/min	This line shows the flow rate of the carrier gas passing through the column. Press ON to display the actual and setpoint values. Press OFF or 0 to turn off all inlet pressures.
`Lin. veloc.`	Not editable	This line shows the velocity of the carrier gas through the column, expressed in cm/s.
`Void time`	Not editable	This line indicates the elution time, expressed in seconds, of an un-retained peak.
`Flow mode`	`Prog flow`	This indicates the selected flow mode.

Table 4-4. Carrier Menu in Programmed Flow Mode (Continued)

Menu	Range	Comments
`Initial flow`	0.0–100 mL/min	This line defines the beginning flow rate.
`Initial time`	0–999.99 min	This line defines the length of time the GC maintains the `Initial flow`.
`Ramp 1`	On/Off, ∞, 0–120 mL/min	This line defines the ramp rate in mL/min to reach the *final flow rate*. Press ON to enable the ramp and display the setpoint value.
`Final flow`	0–100 mL/min	This parameter defines the final flow rate the carrier gas will reach at the end of the ramp rate.
`Final time`	0–999.99 min, ∞	This parameter defines how long the corresponding `Final flow` must be kept.
`Ramp 2-3`	On/Off, ∞, 0–120 mL/min^2	To program additional ramps, press ON and enter the ramp rates in mL/min^2. 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`.
`Gas Saver Flow`[1]	On/Off, 0–500 mL/min	This line indicates the gas saver flow. Press ON to turn on the gas saver flow and to display the setpoint value. Press OFF to turn off the gas saver flow. The flow is retained in memory.
`Saver time`[1]	0.00–999.99 min	This line shows the gas saver time, which is the time in the run at which the gas saver function starts to operate. This line does not appear if `Gas saver flow` is `Off`.
`Vacuum comp`	On/Off	Use this parameter only when the TRACE GC Ultra is used with a mass detector to compensate for vacuum column outlet.

1. This parameter is displayed only for the S/SL and PTV injectors.

Programmed Pressure Mode

In programmed pressure mode, the inlet pressure can be programmed to change during the analytical run. In this mode, up to three pressure ramps can be entered.

Table 4-5. Carrier Menu in Programmed Pressure Mode

Menu	Range	Comments
LEFT CARRIER		This line is the menu title bar.
Pressure	On/Off, 2–250 kPa or 10–1000 kPa[1]	This line shows the constant pressure of the carrier gas passing through the column. Press ON to display the actual and setpoint values. Press OFF or 0 to turn off all inlet pressures.
Col. flow	Not editable	This line displays the actual column flow value that depends on the pressure set. This parameter is not editable in programmed pressure mode.
Lin. veloc.	Not editable	This line shows the velocity of the carrier gas through the column, expressed in cm/s.
Void time	Not editable	This line indicates the elution time, expressed in seconds, of an un-retained peak.
Flow mode	Prog Pres	This line indicates the selected flow mode.
Initial Pressure	2–250 kPa or 10–1000 kPa	This line defines the initial pressure.
Initial time	0–999.99 min,∞	This line defines the length of time the GC maintains the initial pressure.
Ramp 1	On/Off, ∞, 0–120 kPa/min	This line defines the ramp pressure in kPa/min to reach the Final pressure. Press ON to enable the ramp and display the setpoint value.
Final pressure	2–250 kPa or 10–1000 kPa[1]	This parameter defines the final pressure the carrier gas will reach at the end of the ramp rate. This line does not appear unless a ramp has been activated.
Final time	0–999.99 min, ∞	This parameter defines how long the corresponding *final pressure* must be maintained. This line does not appear unless a ramp has been activated.

Table 4-5. Carrier Menu in Programmed Pressure Mode (Continued)

Menu	Range	Comments
`Ramp 2-3`	On/Off, ∞, 0–120 kPa/min	To program additional ramps, press 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`.
`Gas Saver Flow`[2]	On/Off, 0–500 mL/min	This line indicates the gas saver flow. Press ON to turn on the gas saver flow and display the setpoint values. Press OFF to turn off the gas saver flow. The flow is retained in memory.
`Saver time`[2]	0–999.99 min	This line shows the gas saver time, which is the time in the run at which the gas saver function starts to operate. This line does not appear if `Gas saver flow` is `Off`.
`Vacuum comp`	On/Off	Use this parameter only when the TRACE GC Ultra is used with a mass detector to compensate for vacuum column outlet.

1. The default pressure unit is kPa. You can change the units to psi or bar in the **CONFIGURE** menu.
2. This parameter is displayed only for the S/SL and PTV injectors.

OPERATING SEQUENCE

Configuring the Carrier Gas

To change the carrier gas configuration, proceed as follows:

1. Press CONFIG and scroll to `Left carrier` or `Right carrier`.
2. Press MODE/TYPE to display a submenu of carrier gases.

```
     CONFIG LEFT CARRIER
* Helium                    <
  Hydrogen
  Nitrogen
  Ar/CH4  5%
  Argon
```

3. Scroll to the gas to be used and press ENTER to confirm the selection. An asterisk appears on the left of the gas selected.

NOTE

Hydrogen will always be displayed, but it can be selected only if a hydrogen sensor is installed in the column oven. If not, the message **`Hydrogen sensor required`** will be displayed if you try to select hydrogen.

OPERATING SEQUENCE

Programming the Carrier Gas Parameters

Before you begin this procedure, do the following:

- Check that the carrier gas type is correct for the analysis.

When you install a new column, you must perform a column evaluation.

- Press LEFT CARRIER or RIGHT CARRIER to open the appropriate **CARRIER** menu.

Select the Carrier Flow Mode

1. Scroll to `Flow mode` and press MODE/TYPE or ENTER.
2. Scroll to the mode you want and press ENTER.

Enter the Initial Flow or Pressure

1. If you selected `Constant flow` mode, scroll to `Col. flow` and enter the desired initial value. Press ENTER. The GC calculates the pressure necessary and adjusts the pressure as necessary to maintain the constant flow.
2. If you selected `Constant pressure` mode, scroll to `Pressure` and enter the desired initial value. Press ENTER.

Enter a Flow or Pressure Program

When you select programmed flow or programmed pressure, the **CARRIER** menu contains parameters for up to three program ramps.

1. If you selected `Prog flow`, scroll to `Initial flow` and enter the desired value. Press ENTER.
2. If you selected `Prog pressure`, scroll to `Pressure` and enter the desired value. Press ENTER.
3. Scroll to `Initial time` and enter a value. This parameter ends the initial part of the program.

Program the Ramps

1. To program a ramp, scroll to `Ramp 1` and enter the value.
2. Scroll to `Final flow 1` or `Final pres 1` and enter the final value for the ramp.
3. Scroll to `Final time 1` and enter the final time for Ramp 1. This operation ends the first ramp setting.
4. 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.
5. Scroll to `Final flow 2` or `Final pres 2` and enter the final value for the ramp.
6. Scroll to `Final time 2` and enter the final time for Ramp 2. This operation ends the second ramp setting.
7. 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.
8. Scroll to `Final flow 3` or `Final pres 3` and enter the final value for the ramp.
9. Scroll to `Final time 3` and enter the final time for Ramp 3. This operation ends the third ramp setting.

SECTION

III

Injectors

This section contains information about the injection systems available for the TRACE GC Ultra.

Chapter 5, *Split/Splitless Injector (S/SL)*, describes the split/splitless injector and contains operating procedures for the different split/splitless operating modes.

Chapter 6, *On-Column Injector (OCI)*, describes the on-column injector, on-column injection techniques, and operating procedures.

Chapter 7, *High Oven Temperature Cold On-Column Injector (HOT OC)*, describes the HOT on-column injector for injections at high oven temperatures, HOT on-column injection techniques, and operating procedures.

Chapter 8, *Large Volume On-Column Injector (LVOCI)*, describes the on-column injector used for large volume injections with an autosampler.

Chapter 9, *Packed Column Injector (PKD)*, describes the packed column injector and explains the packed column operating procedures.

Chapter 10, *Purged Packed Column Injector (PPKD)*, describes packed column injectors with a septum purge. Included in this chapter are injection techniques and operating procedure descriptions.

Chapter 11, *Programmable Temperature Vaporizing Injector (PTV)*, describes the Programmable Temperature Vaporizing injector and

contains operating procedures for using the injector in different operating modes.

Chapter 12, *Gas Sampling Valve (GSV)*, describes the gas sampling valves available with the TRACE GC Ultra and contains operating sequences for manual and automatic sampling.

5

Split/Splitless Injector (S/SL)

This chapter describes the Split/Splitless (S/SL) injector and contains operating sequences for the different split/splitless operating modes.

Chapter at a Glance…

S/SL Overview 106
S/SL Injection Techniques 116
SSL Backflush Operation 122
Large Volume Splitless Injector (LVSL) 124
S/SL Injector Menus 128

Operating Sequences

Installing a Liner and Septum 132
Programming the Split Mode 133
Programming the Splitless Mode 134
Programming the Surge Splitless Mode 135
Programming the Large Volume Splitless Method 136
Performing a S/SL Injection 139
Performing a LVSL Injection 141

S/SL Overview

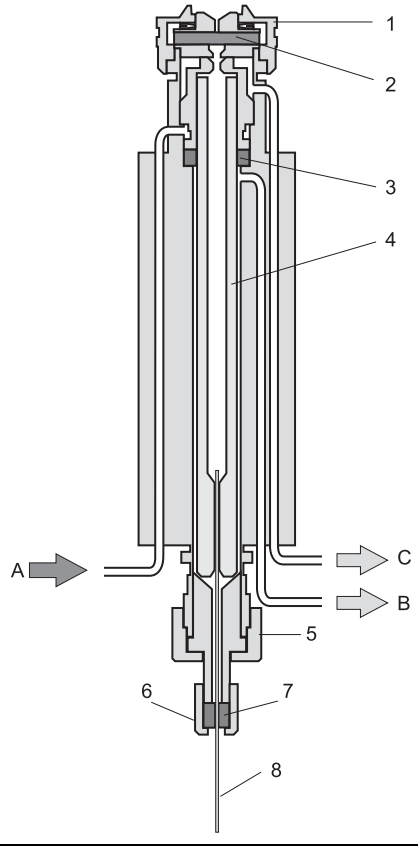
The S/SL injector, shown in Figure 5-1, is optimized for either *split* or *splitless* applications to ensure effective sample transfer into the column, minimizing heavy component discrimination. For both split and splitless applications, the sample is injected through a septum into a glass liner in the vaporization chamber. The technique used, either split or splitless, determines the choice of the glass liner and the length of the syringe needle. You can control the injector temperature from ambient to 400 °C, although the actual injector temperature you use depends on the solvent choice and thermal stability of the samples.

An electronic device controls the split flow, while the septum purge flow is kept constant by a calibrated flow regulator. The S/SL injector is also equipped with electronically actuated On/Off valve for septum purge line. Volatile components given off by the hot septum can produce ghost peaks in a chromatogram. The septum purge system can continually purge the septum with a flow of gas. This prevents the volatile components given off by the septum from entering the column. Figure 5-2 shows the septum purge system. Figure 5-3 shows the S/SL injector components.

Large Volume Splitless Injector (LVSL)

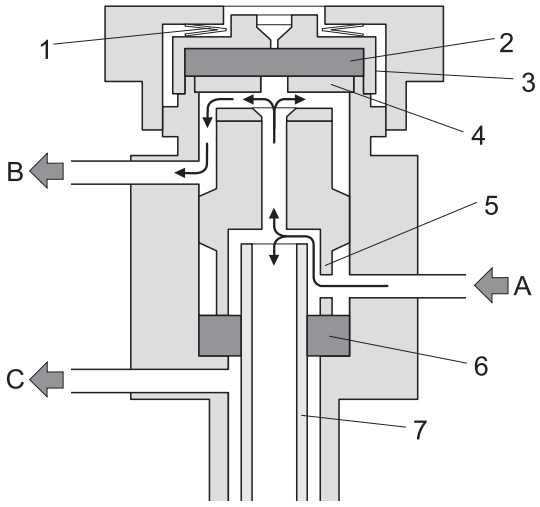
A special setup of the split/splitless injector that allows the introduction of large sample volumes. Refer to *Large Volume Splitless Injector (LVSL)*.

The use of a dedicated software named **LVSL Assistant** is used to determine the best temperature and pressure settings for the LVSL injection. Besides, it allows to estimate the shortest oven initial time in relation to the chosen large volume, kind of solvent and other parameters like carrier and inlet conditions.



1. Septum Cap	7. Graphite Column Ferrule
2. Septum	8. Capillary Column
3. Graphite Liner Seal	A. Carrier Gas Inlet
4. Glass Liner	B. Split Line
5. Fixing Nut	C. Septum Purge Line
6. M4 Retaining Nut	

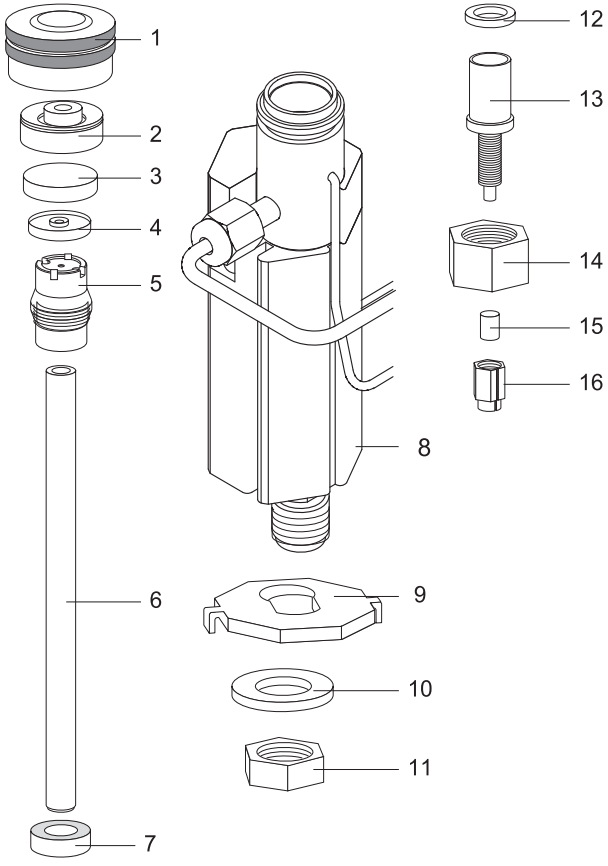
Figure 5-1. Split/Splitless Injector



1. Spring	6. Graphite Liner Seal
2. Septum	7. Glass Liner
3. Septum Holder	A. Carrier Gas Inlet
4. Septum Support	B. Septum Purge Line
5. Liner Cap	C. Split Line

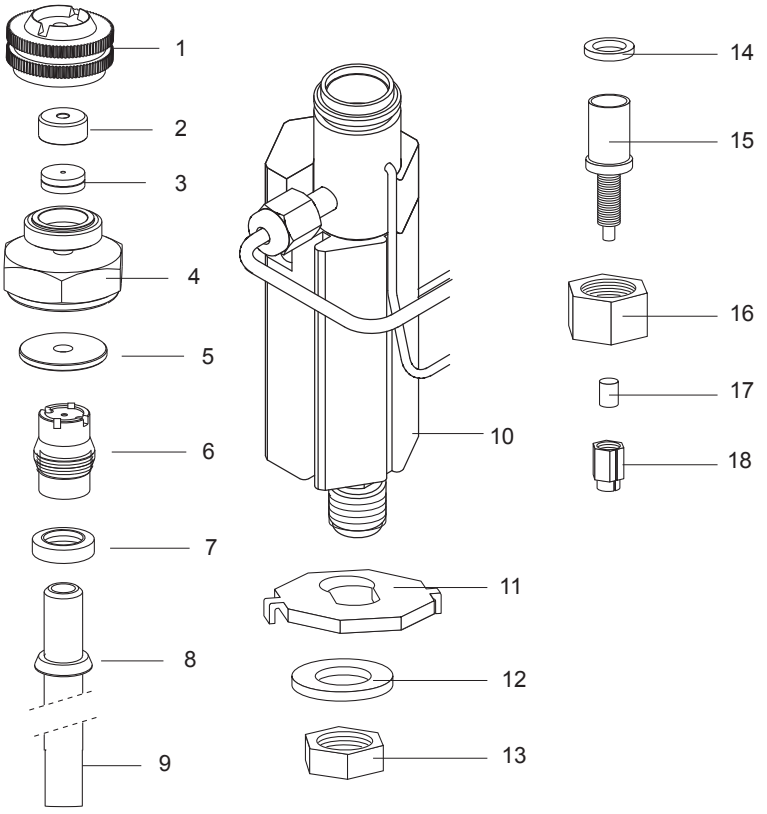
Figure 5-2. Septum Purge System

Figures 5-3 and 5-4 shown the components of the split/splitless injector and large volume splitless injector respectively.



1. Septum Cap	9. Anti-rotation Plate
2. Septum Holder	10. Metal Ring
3. Septum	11. Fixing Nut
4. Septum Support	12. Silver Seal
5. Liner Cap	13. Terminal Fitting
6. Liner	14. Fixing Nut
7. Graphite Liner Seal	15. Graphite Column Ferrule
8. Injector Body	16. M4 Retaining Nut

Figure 5-3. Split/Splitless Injector Components



1. Septum Cap	10. Injector Body
2. Septum Holder	11. Anti-rotation Plate
3. 9-mm Septum	12. Metal Ring
4. LVSL Adapter	13. Fixing Nut
5. LVSL Adapter Vespel Seal	14. Silver Seal
6. Liner Cap	15. Terminal Fitting
7. Metal Ring	16. Fixing Nut
8. Vespel Liner Seal (already installed)	17. Graphite Column Ferrule
9. Liner	18. M4 Retaining Nut

Figure 5-4. Large Volume Splitless Injector Components

Septum

Standard Septum

You should always use good quality septa, such as the BTO septa supplied with the TRACE GC Ultra. Such septa resist deformation, have longer life expectancy, and have a low bleed level, even at high temperatures.

Microseal™ Valve

S/SL injector is compatible with the use Merlin Microseal™ High Pressure Valve instead of the standard septa.

NOTE To replace the standard septum with the Microseal™ Valve, the relevant installation kit is required.

Microseal™ valve requires a 0.63 mm diameter (0.025-inch) blunt tip syringe or the side hole needle tip.

Liners

You may install different types of glass liners depending on the injection mode used. Figure 5-5 and Table 5-1 shown the liner options.

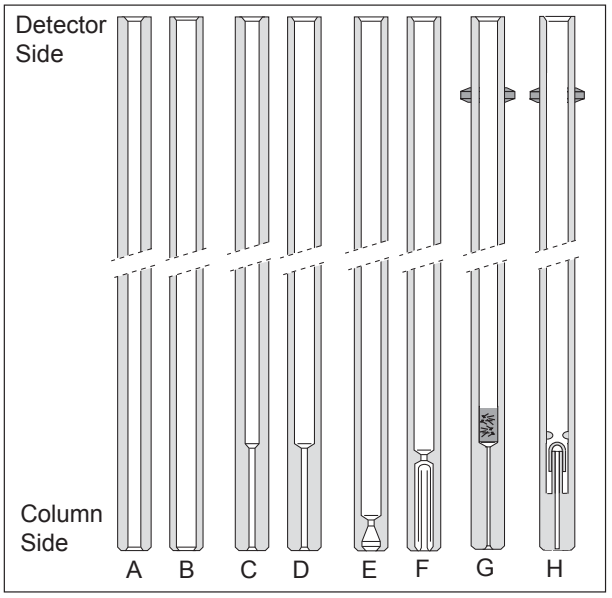
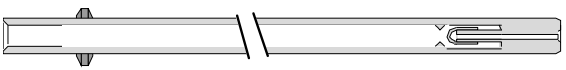


Figure 5-5. Injector Liners

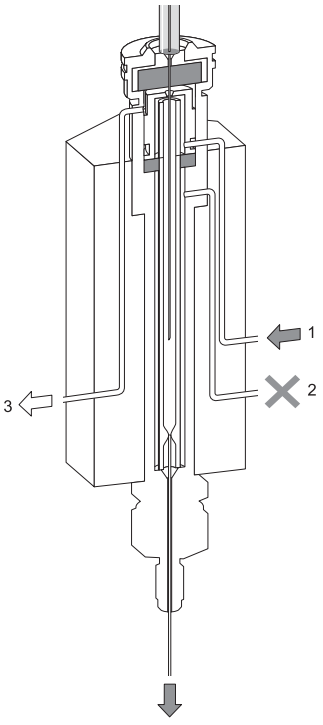
Table 5-1. Injector Liner Sizes and Applications

ID#	Part Number	Liner Type Description and Application
A	453 200 31	 3 mm ID; 8 mm OD; 105 mm length. Glass liner used for split injections.
B	453 200 30	 5 mm ID; 8 mm OD; 105 mm length. .Glass liner used for split injections.

Table 5-1. Injector Liner Sizes and Applications (Continued)

ID#	Part Number	Liner Type Description and Application
C	453 200 32	 3 mm ID; 8 mm OD; 105 mm length. Glass liner used for splitless injections.
D	453 200 33	 5 mm ID; 8 mm OD; 105 mm length. Glass liner used for splitless injections.
E	453 003 10	 5 mm ID; 8 mm OD; 105 mm length. Glass liner used for direct injections into a wide-bore column.
F	453 003 20	 5 mm ID; 8 mm OD; 105 mm length. Glass liner used for split injections at high flow rates or for the most polar solvents.
G	453 020 65	 5 mm ID; 8 mm OD; 105 mm length. Glass liner packed with deactivated glass wool used for large volume splitless injection of dirty sample (Vespel seal included).
H	453 220 67	 5 mm ID; 8 mm OD; 105 mm length. Deactivated laminar liner used for large volume splitless injection of samples containing labile compounds (Vespel seal included).

The glass liner used for direct splitless injection into a wide-bore column is tapered at the bottom. It is used with 0.53 mm ID columns. Figure 5-6 shows the tapered glass liner.



1. Carrier Gas Inlet
2. Split Line
3. Septum Purge Line

Figure 5-6. S/SL Wide-Bore Injection with a Tapered Liner

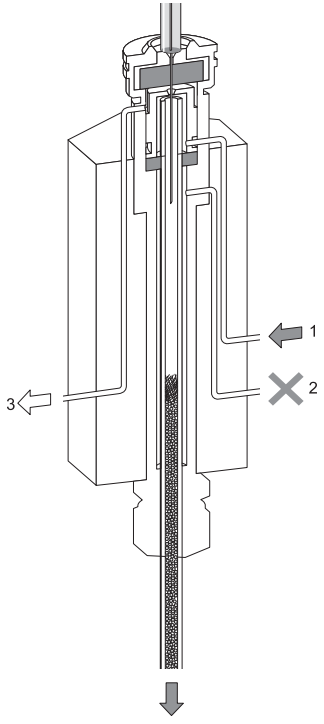
A laminar cup liner is used for split injections at high split flow rates or for the more polar solvents. This glass liner has a mixing chamber with an extended flow path that allows complete sample vaporization before the sample reaches the split point.
LV-liner packed with glass wool on the bottom is used for large volume splitless injections. Please note that LV liners have already mounted a vespel seal and a

metallic back ferrule to ensure perfect tightness requested when the liner is filled with large amounts of vapor.

LV-Laminar cup liner should be used when analyzing labile substances which can suffer breakdown on high surface packing.

Packed Columns

With a special conversion kit, you can install packed columns in the S/SL injector, as shown in Figure 5-7.



1. Carrier Gas Inlet
2. Split Line
3. Septum Purge Line

Figure 5-7. S/SL with a Packed Column

S/SL Injection Techniques

You use different sample injection techniques for split and splitless applications.

Split Injection Technique

In split injection, 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. Figure 5-8 illustrates the gas flows for the split injection technique.

You inject the sample into a glass liner inside the heated vaporization chamber. In the chamber, the sample undergoes rapid vaporization. The relatively high gas flow through the injector carries the vaporized sample rapidly down toward the head of the column.

At the column head, the sample splits in the split ratio. A portion of the sample goes into the column, while the remainder is carried out the split line. You set the column flow and the split flow in the **`LEFT`** or **`RIGHT INLET`** menu.

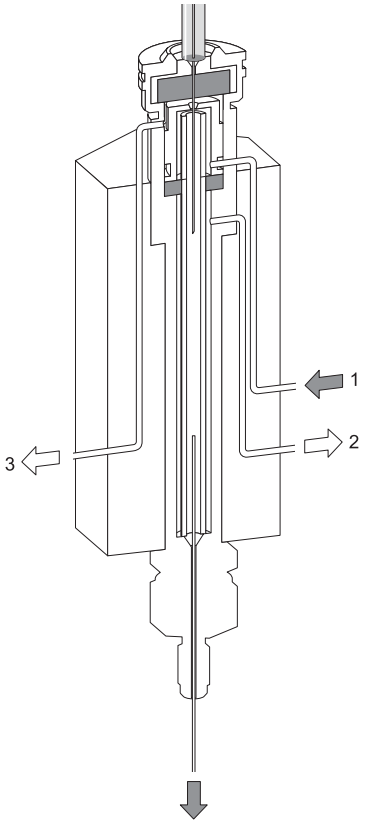
Narrow bore columns, which have inherently low column flows, can produce relatively high split ratios.

NOTE

Hot Empty Needle Injection Technique

Using conventional syringes in hot injectors may cause discrimination of higher boiling point components. This is due to partial sample vaporization within the hot syringe needle. We recommend you use a *hot empty needle* injection technique. This technique consists of drawing the sample volume into the syringe barrel followed by a small air gap, which ensures the syringe needle is empty. You insert the empty needle into the injector, wait a few seconds, inject the sample rapidly, and immediately remove the syringe.

Split injection is suitable for high-concentration sample analysis, headspace analysis, and isothermal analysis.



1. Carrier Gas Inlet
2. Split Line
3. Septum Purge Line

Figure 5-8. Split Injection Technique

The major advantages of split injection are simplicity and the ability to introduce samples over a wide range of concentrations. Peak shapes in the chromatogram are generally very sharp due to the rapid sample introduction into the column.

Splitless Injection Technique

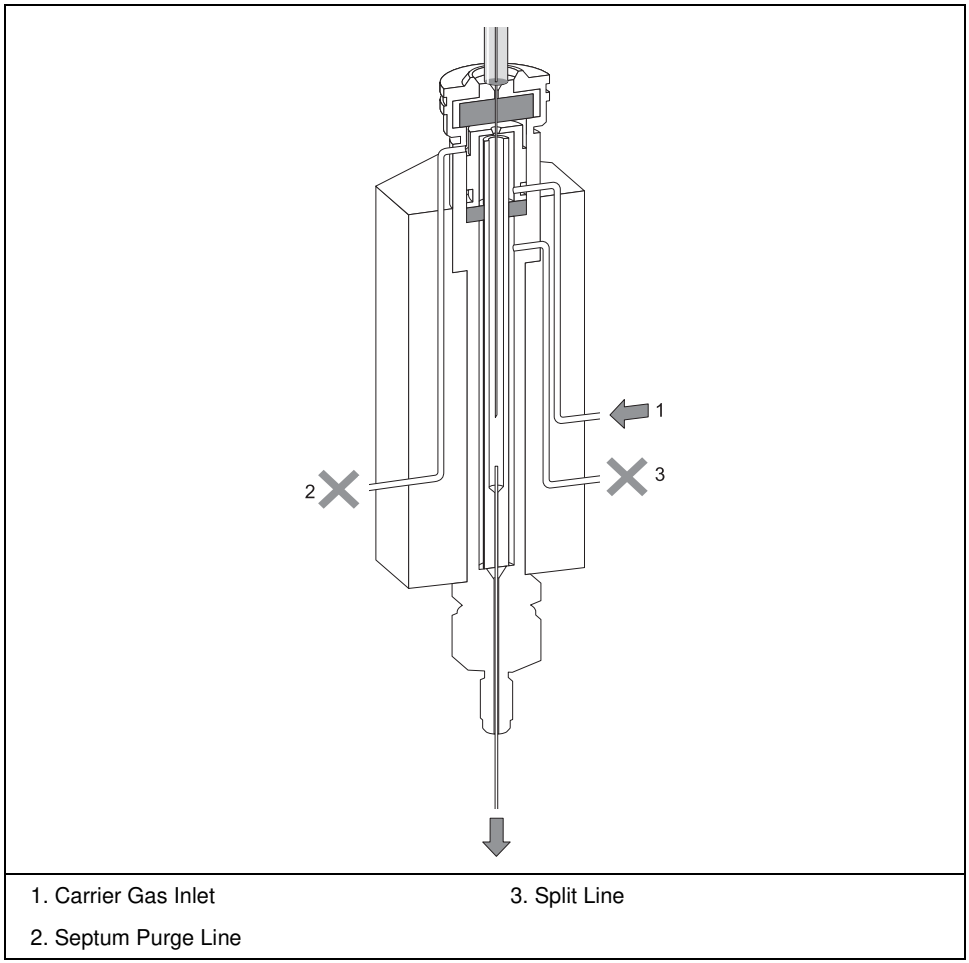
Splitless injection is suitable for the analysis of compounds present in very low concentrations and for relatively dirty matrices.

The splitless technique allows the entire sample to enter the column without splitting. This offers better sensitivity than the split technique. Compared to on-column injection, which is also suitable for capillary column analysis of compounds at low concentrations, splitless injection has the major advantage that it can accommodate significantly dirty samples.

With splitless injection, the split line is closed during 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. Figure 5-9 shows the split/splitless injector used for splitless injection.

During splitless injection, when the split valves are closed, the flow of gas through the injector is relatively low. It is equal to the column flow—only a few mL/min.

The vapor cloud generated by the vaporization of the liquid sample expands upward from the point of vaporization and can fill the liner.
The injector can accept and quantitatively transfer to the column sample volumes of up to 5 µL.
With injection volumes higher than 4 µL, the recovery of the sample injected can be improved by closing the septum purge as well as the split valve during the splitless period.
You can program this in the **INLET** menu when you select the `Splitless` mode.
Condensation and subsequent loss of higher molecular mass compounds in the top region of the injector liner is prevented by effective heating over the whole length of the injector.



1. Carrier Gas Inlet
2. Septum Purge Line
3. Split Line

Figure 5-9. Splitless Injection Technique

The transfer of the vaporized sample from the injector to the column takes place very slowly due to the relatively low column flows involved. With typical carrier gas flow rates of 1–4 mL/min, the transfer can take between 30 and 90 seconds, depending on a variety of circumstances.

This transfer time is the *splitless time*. You can set the splitless time in the **INLET** menu when you select `Splitless` mode. For narrower diameter columns (< 0.22 mm) with inherently lower flows (< 1.0 mL/min), the transfer might never be

completely achieved due to back diffusion of sample vapors in the injector at a higher rate than transfer into the column.
You can counter this by using the *splitless surge* pressure mode. In this mode, the pressure in the injector temporarily increases during the splitless period to increase the flow into the column. You set the surge pressure, which activates during the **Prep Run** stage.

At the end of the splitless period, the split valve reopens and the split flow flushes the injector of any remaining sample vapors. In splitless injection, the absolute split flow is not important. It need only be sufficient to purge the injector. Normally 40–50 mL/min is adequate.

Refocusing the Sample

The sample vapors enter the column over an extended period of time and produce very broad starting bands. To maintain column efficiency, some form of refocusing must take place in the column inlet before chromatography begins. To achieve this, keep the oven temperature to a sufficiently low value during the transfer of the sample to trap it on the column head by condensation or solvent effect.

This technique's efficiency is greatly enhanced by correctly choosing conditions for column character, carrier gas flow rates, splitless time, column temperature, and injector liner internal diameter. All of these conditions can affect the transfer efficiency and refocusing.

- **Solvent Effect**
  To refocusing the compounds that elute at low temperature, the so-called *solvent effect* is used. It consists of the volatile compounds trapping on the solvent recondensed in column. It is obtained cooling the column to 20–25 °C below the solvent boiling point, combined with injection volumes of at least 1 µL. Isothermal analysis or temperature programming can then continue. You must carefully control the analysis conditions and use a 7 cm syringe needle applying the *Hot Empty Needle Injection Technique*.
- **Temperature Effect**
  You can refocus later eluting compounds without solvent effects by cooling the oven sufficiently during the transfer. The trapping temperature effect traps and refocuses the sample compounds.

Flooding

Splitless injections may occasionally exhibit an effect known as *flooding*, which can result in peak distortion due to the solvent condensation. You can overcome flooding effects by using a *retention gap*. Refer to *Retention Gaps/Pre-Columns* in Chapter 6, *High Oven Temperature Cold On-Column Injector (HOT OC)*, for more information.

NOTE

Hot Empty Needle Injection Technique

Using conventional syringes in hot injectors may cause discrimination of higher boiling point components. This is due to partial sample vaporization within the hot syringe needle. We recommend you use a *hot empty needle* injection technique. This technique consists of drawing the sample volume into the syringe barrel followed by a small air gap, which ensures the syringe needle is empty. You insert the empty needle into the injector, wait a few seconds, inject the sample rapidly, and immediately remove the syringe.

SSL Backflush Operation

With the implementation of the backflush kit the TRACE GC Ultra equipped with the SSL injector, will be able to perform operations with the following advantages:

With the implementation of the Backflush kit, the TRACE GC Ultra equipped with the SSL injector, will be able to perform operations with the following advantages:

- Eliminate during the cleaning phase the heavy part of the sample, which are not relevant for the analysis. This will strongly reduce the analysis time with any analytical set-up and with many samples.

  This step is important when performing analysis of volatile compounds in a relatively low volatile mixture.
- Avoid solvent introduction into the column when performing a large volume injection. This is particularly important with MS applications.
- Perform precise cuts of the chromatogram, installing a selected coated precolumn, so that only a part of the sample is transferred into the column for the analysis.
- Use of very narrow bore column without significant peak broadening effect.

  In this way, for example, it is possible to use a thick film of stationary phase and to perform a precise cut of the components that are not of interest, so that is possible to analyze only the volatile compounds even with narrow bore capillary columns.

  The rest of the sample is eliminated through the injector and the oven temperature does not need to be increased to elevated value.

NOTE To install and configure the Backflush option refer to the Backflush System for SSL Injector Installation Guide.

The Backflush principle of operation is schematically shown in Figure 5-10.

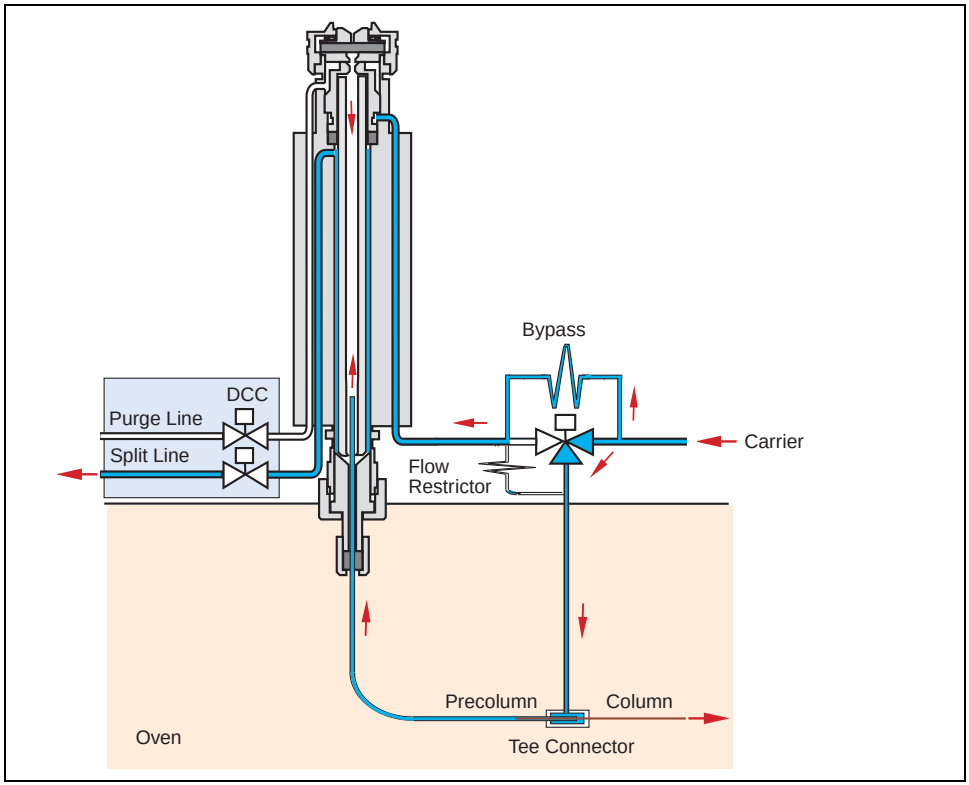
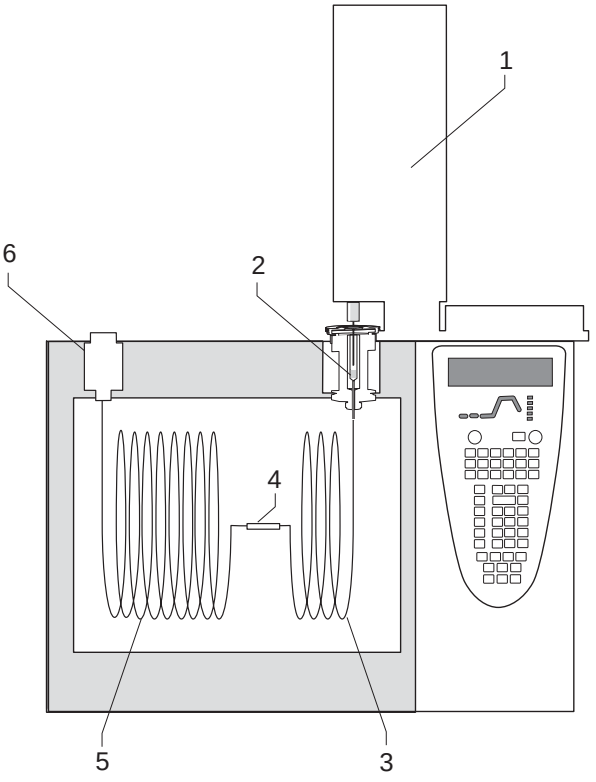


Figure 5-10. Backflush System for SSL Injector

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 AS or AI/AS 3000 autosampler.



1. Autosampler	2. Liner with Glass Wool
3. Pre-column	4. Press-fit Connection
5. Analytical Column	6. Detector

Figure 5-11. Configuration for Large Volume Splitless Injection

LV Splitless Injection Requirements

Large Volume-Splitless injection requires:

- LVI Analytical Kit for SSL (PN 19050243) including:
  - — Kit Assy Split Line 1/16” (PN 19050710)
  - — Kit Assy Dummy Filters (PN 19050690)
- a dedicated liner already equipped with its own Vespel ferrule:
  - — liner packed with deactivated glass wool (standard)
  - — or deactivated laminar cup liner (optional)
- an uncoated pre-column with a capacity for retaining an amount of liquid at least corresponding to the volume of sample injected (e.g. 5 m x 0.32 mm i.d. or 3 m x 0.53 mm i.d. for 30 µl volumes).

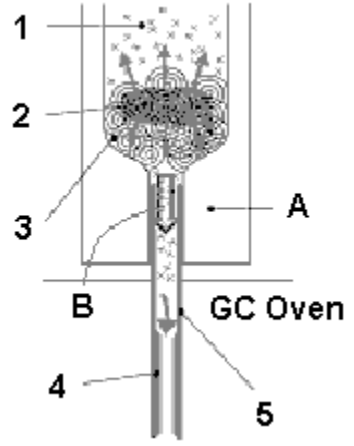
To correctly perform the Large Volume Splitless injection be sure to replace the filters on the carrier and split lines with the provided dummy filters included into the LVI Analytical Kit for SSL.

The LV Splitless Injection Technique

In the standard splitless mode the injector can accept and quantitatively transfer to the column sample volumes of up to 5 µl. Splitless injections of higher amounts are possible by the Concurrent Solvent Recondensation technique (CSR LVSL). The LVSL technique can be summarized by five key steps.

1. Injection with liquid band formation
2. auto pressure surge
3. recondensation of the solvent vapors in the pre-column concurrent with solvent evaporation in the injector
4. solutes transfer
5. solvent evaporation in the pre-column

The mechanism of the concurrent solvent recondensation is shown in Figure 5-12



1. Solvent Vapors	2. Liquid Sample
3. Glass Wool	4. Recondensed Solvent
5. Pre-column	A. Liner Bottom
B. High Flow	

Figure 5-12. 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.

S/SL Injector Menus

The **INLET (S/SL)** menu includes the operating parameters for the split/splitless injector. The parameters you can edit depend on the operating mode chosen: split, splitless, or splitless with surge.

Press LEFT INLET or RIGHT INLET to display the **LEFT** or **RIGHT INLET (S/SL)** menu.

```
      LEFT INLET (S/SL)
 Temp              250  250
 Pressure       10.6   10.6
 Mode:                split<
```

The `Mode:` menu item displays the current operating mode.

Press MODE/TYPE to open the **INLET MODE** submenu.

```
        XX INLET MODE
* Split                  <
  Splitless
  Splitless w/surge
```

Scroll to the mode you want to use and press ENTER to confirm the selection. An asterisk appears on the left of the operating mode selected.

Tables 5-2 through 5-4 explain the ranges and functions of the parameters in the **LEFT** and **RIGHT INLET (S/SL)** menus for each of the four operating modes.

The injector and carrier gas menus are related. If you set a pressure in the carrier gas menu, that same pressure setting is reflected in the injector menu and vice-versa.

The items in the inlet menu vary depending on the operating mode you select in the **LEFT** or **RIGHT INLET MODE** menu. Tables 5-2 through 5-4 show the split/splitless inlet menu for the operating modes.

Table 5-2. Inlet (S/SL) Menu in Split Mode

Menu	Range	Comments
RIGHT INLET (S/SL)		This line is the menu title bar.
Temp	On/Off, 0–400 °C	This line shows the base injector temperature. Press ON to turn on the heater and to display the actual and setpoint values. Press OFF to turn off the heater and display the actual value.
Pressure	On/Off, 2–250 kPa or 10–1000 kPa[1]	This line shows the pressure. Press ON to display the actual and setpoint values. Press OFF or 0 to turn off all inlet flows and display the actual value.
Mode: Split		This line displays the inlet operating mode selected.
Total flow	Not editable	This line shows the total gas flow consumption, which is the sum of the column flow, split flow (or gas saver flow), and septum purge flow.
Split flow	On/Off, 0, 10–500 mL/min	This line shows the split flow. Press ON to turn on the split flow and display the actual and setpoint values. Press OFF or 0 to close the split valve and to turn off the split flow.
Split ratio	1–5000	This line displays the actual value of the split ratio. This value is the ratio between the split flow and the column flow.

1. 0.3–36 psi, 0.02–2.5 bar; 0.145–145 psi, 0.1–10.00 bar.

Table 5-3. Inlet (S/SL) Menu in Splitless Mode

Menu	Range	Comments
RIGHT INLET (S/SL)		This line is the menu title bar.
Temp	On/Off, 0–400 °C	This line shows the base injector temperature. Press ON to turn on the heater and to display the actual and setpoint values. Press OFF to turn off the heater and to display the actual value.

Table 5-3. Inlet (S/SL) Menu in Splitless Mode (Continued)

Menu	Range	Comments
`Pressure`[2]	On/Off, 2–250 kPa or 10–1000 kPa[1]	This line shows the pressure. Press ON to display the actual and setpoint values. Press OFF or 0 to display the actual value and to turn off inlet pressure, thereby turning off the flow.
`Mode: Splitless`		This line displays the operating mode selected. Press ENTER or MODE/TYPE to change the operating mode.
`Total flow`	Not editable	This line shows the total gas flow consumption, which is the sum of the column flow, split flow (or gas saver flow), and septum purge flow.
`Split flow`	On/Off, 0, 10–500 mL/min	This line shows the split flow. Press ON to turn on the split flow and to display the actual and setpoint values. Press OFF or 0 to close the split valve and to turn off the split flow.
`Splitless time`	0–999.99 min	This line shows the splitless time, which is the duration of split valve closure.
`Const sept purge?`	Yes/No	Press YES to activate a constant septum purge to continuously flush the septum with a purge flow of 5 mL/min when using helium or nitrogen as a carrier gas or 10 mL/min when using hydrogen as a carrier gas.
`Stop purge for:`	0–999.99 min, ∞	This line appears only when `Constant septum purge` is set to `No`.

1. 0.3–36 psi, 0.02–2.5 bar; 0.145–145 psi, 0.10–10.00 bar.

Table 5-4. Inlet (S/SL) Menu in Surge Splitless Mode

Menu	Range	Comments
`RIGHT INLET (S/SL)`		This line is the menu title bar.
`Temp`	On/Off, 0–400 °C	This line shows the base injector temperature. Press ON to turn on the heater and display the actual and setpoint values. Press OFF to turn off the heater and to display the actual value.

Table 5-4. Inlet (S/SL) Menu in Surge Splitless Mode (Continued)

Menu	Range	Comments
Pressure	On/Off, 2–250 kPa, 10–1000 kPa[1]	This line shows the pressure. Press ON to display the actual and setpoint values. Press OFF or 0 to display the actual value and to turn off inlet pressure, thereby turning off the flow.
Mode: SRG Splitless		This line displays the operating mode selected. Press ENTER or MODE/TYPE to change the operating mode.
Total flow	Not editable	This line shows the total gas flow consumption, which is the sum of the column flow, split flow (or gas saver flow), and septum purge flow.
Split flow	On/Off, 0, 10–500 mL/min	This line shows the split flow. Press ON to turn on the split flow and display the actual and setpoint values. Press OFF or 0 to close the split valve and to turn off the split flow.
Splitless time	0–999.99 min	This line shows the splitless time, which is the duration of split valve closure.
Surge pressure	2–250 kPa or 10–1000 kPa[1]	This line indicates the surge pressure, which is activated at **Prep Run**.
Surge duration	0–999.99 min	This line indicates the duration of the surge pressure after run start.
Const sept purge?	Yes/No	Press YES to activate a constant septum purge to continuously flush the septum with a purge flow of 5 mL/min when using helium or nitrogen as a carrier gas or 10 mL/min when using hydrogen as a carrier gas.
Stop purge for:	0–999.99 min, ∞	This line appears only when Constant septum purge is set to No.

1. 0.3–36 psi, 0.02–2.5 bar; 0.145–145 psi, 0.10–10.00 bar.

OPERATING SEQUENCE

Installing a Liner and Septum

Materials required:

- liner
- septum
- spacer
- tweezers
- graphite seal
- screwdriver

WARNING! The injector fittings may be hot.
This sequence must be performed with the injector at room temperature.

1. Choose the correct liner for your application (see Table 5-1 on page 112). Slide a graphite seal onto the liner while gently turning the seal. Push it to 8–10 mm from the top of the liner.

CAUTION Be careful not to break the graphite or allow graphite to enter in the liner.

When using LVSL injector the liner is already equipped with the appropriate Vespel liner seal.

2. Holding the top of the liner with tweezers, lower it into the injector. The liner should rest on the spacer at the bottom of the injector.

3. Insert the liner cap and secure it with the screwdriver. The liner cap must be screwed down tight enough to ensure a good seal between the liner and the injector body.

4. Place the septum support in the injector. The septum support must lie flush with the top of the injector. If not, the liner cap may not be tight enough.

5. Use tweezers to pick up the septum. Place the septum into the septum holder, then place the holder on top of the complete injector assembly.

CAUTION Use tweezers to pick up the septum to avoid contaminating it.

6. Gently finger-tighten the septum cap onto the injector assembly to hold the septum in place.

WARNING! Do not overtighten the septum cap. The septum will deform and may be difficult to penetrate with the syringe needle.

OPERATING SEQUENCE

Programming the Split Mode

In split injection, only a portion of the sample transfers to the column. Most of it discharges through the splitting line. The ratio between the split flow and the column flow defines the amount of sample that enters the chromatographic system. The split and column flows must be set to obtain the correct split ratio necessary for the analysis.

Before you begin programming, do the following:

- Verify that a column is correctly installed, the correct liner is in the injector, and the system is free of leaks.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.

1. Press LEFT INLET or RIGHT INLET to open the **`INLET (S/SL)`** menu.
2. Scroll to `Mode:` and press MODE/TYPE.
3. Scroll to `Split` and press ENTER.
4. Scroll to `Temp` and press ON. Set the appropriate value.

WARNING! In the case of DoublePro configuration, the temperature for both injectors must be set at the same value otherwise the GC could not reach the Ready stage.

5. Specify the split flow or the split ratio. To see the split flow, scroll to `Split flow` and enter the value in mL/min. The split ratio will be calculated for you.

   To set the split ratio, scroll to `Split ratio` and enter that value. The split flow will be calculated for you.

OPERATING SEQUENCE

Programming the Splitless Mode

In splitless analyses, the splitting line is closed during the sample transfer onto the column. The time during which the splitting valve remains closed is called the *splitless time*. When the sample transfer ends, the split line reopens to purge the residual sample components, essentially solvent, out of the vaporization chamber. You can activate a constant septum purge, if necessary, to continuously flush the septum with a purge flow. The septum purge prevents septum bleed components from entering the column.

Before you begin programming, do the following:

- Verify that a column is correctly installed, the correct liner is in the injector, and the system is free of leaks.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.

1. Press LEFT INLET or RIGHT INLET to open the **`INLET (S/SL)`** menu.
2. Scroll to `Mode:` and press MODE/TYPE.
3. Scroll to `Splitless` and press ENTER.
4. Scroll to `Temp` and press ON. Enter the appropriate value.

WARNING! In the case of DoublePro configuration, the temperature for both injectors must be set at the same value otherwise the GC could not reach the Ready stage.

5. Scroll to `Split flow` and enter the desired value in mL/min.
6. Scroll to `Splitless time` and enter the time the inlet valve should be closed.

7. If constant septum purge is required, scroll to `Const sept purge?` and press YES to activate a constant septum purge. If constant septum purge is not required, press NO and scroll to `Stop purge for` to enter the time the purge flow should be interrupted.

OPERATING SEQUENCE

Programming the Surge Splitless Mode

In *surge splitless* mode, a carrier gas pressure surge activates during the injection phase for a preset time. This surge accelerates the transfer process of the substances from the injector to the column. The pressure pulse starts in the **Prep Run** phase and ends at the end of the surge duration you program.

Before you begin programming, do the following:

- Verify that a column is correctly installed, the correct liner is in the injector, and the system is free of leaks.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.

1. Press LEFT INLET or RIGHT INLET to open the **`INLET (S/SL)`** menu.
2. Scroll to `Mode:` and press MODE/TYPE.
3. Scroll to `Splitless w/surge` and press ENTER.
4. Scroll to `Temp` and press ON. Enter the appropriate value.

WARNING! In the case of DoublePro configuration, the temperature for both injectors must be set at the same value otherwise the GC could not reach the Ready stage.

5. Scroll to `Split flow` and enter the desired value in mL/min.
6. Scroll to `Splitless time` and enter the time the split valve should be closed.
7. Scroll to `Surge pressure` and enter the value of the pressure surge.

8. Scroll to `Surge duration` and enter the duration of the pressure surge.

9. If constant septum purge is required, scroll to `Const sept purge?` and press YES to activate a constant septum purge. If constant septum purge is not required, press NO and scroll to `Stop purge for` to enter the time the purge flow should be interrupted.

OPERATING SEQUENCE

Programming the Large Volume Splitless Method

A LV Splitless method is programmed in the same way you program standard S/SL methods. No new menus have to be used to prepare a LV analysis. A good starting point is to modify an existing standard splitless method.

Before you begin programming, do the following:

- Verify that the pre-column and the column are correctly installed, the correct liner is in the injector, and the system is free of leaks.

  - Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.
  - Start the *LVSL Assistant* software from data system.

- Estimate the best initial oven condition.

1. First adjust your pre-column settings and enter the K factor obtained in your column evaluation.

2. Select the solvent used for your sample.

3. Decide a volume to inject (up to the Max volume calculated by the Assistant)

4. The Assistant after any changes will update the INITIAL TIME on the bottom at the selected *Initial Oven temp.*

If the calculated *INITIAL TIME* is considered too long (e.g. above 10 minutes) it can reduced by using an higher *Initial Oven temp* provided it is below the *Max Initial Oven Temp*.

If the calculated *INITIAL TIME* is still too long you can chance your pressure settings. This will increase the *Max Initial Oven Temp* parameters letting you choose a higher *Initial Oven temp*.

Example:

Supposing you want to inject 30 µl in Cyclohexane in a system with K factor 1.5, column flow of 2 mL/min with Nitrogen as carrier and a 5 m length, 0.32 mm ID pre-column.

a. Enter 30 in the *Injection Volume* cell

b. Select Cyclohexane in the *Solvent* cell.

c. Select *Constant Flow* in the *Options/Flow mode* menu. This will lets you enter 2 in the *Column Flow* cell.

d. Enter 1.5 in the *K factor* cell.

e. Select Nitrogen in the *Carrier gas* cell.

f. Enter 70 in the *Initial Oven temp* cell.

g. The Assistant will calculate about 9 min *INITIAL TIME*

h. Now enter 80 in the *Initial Oven temp* cell.
The Assistant will calculate about 6 min *INITIAL TIME*

i. Now enter 90 in the *Initial Oven temp* cell.
The Assistant will calculate about 4 min *INITIAL TIME*

j. Now enter 2.5 in the *Column flow* cell.
The Assistant will take the *INITIAL TIME* below 5 min.

This shows that the *INITIAL TIME* can be strongly reduced (even halved) by a proper selection of the inlet and oven parameters. For a full description on the use of the Assistant see *Contents* in the *Assistant Help File*.

Creating a LVSL Method

Set the required parameters as the following guideline:

Parameter	Set
Oven	
Initial oven temp	The value chosen in the Assistant page.
Initial hold time	The value calculated in the Assistant page.
Inlet	
Mode:	Splitless
Temp	An appropriate value in relation to the sample as used in conventional splitless
Split flow	Equal or higher than 50 mL/min
Splitless time	Equal or higher than 0.8 minutes
Constant septum purge	Off
Stop purge time	0.8 minutes
Carrier	
Flow mode	The value selected in Assistant page
Pressure/Flow	The value chosen in the Assistant page
NOTE Any other parameter (detector conditions included) can be maintained as in the pre-existent standard splitless method.	
Autosampler	
Sample volume	The value chosen in the Assistant page
Filling volume	About half the value of the sample injection volume
Air volume	2 µL
Inject delay	0 sec
Pull out delay	0 sec
Sample pull-up speed	5 µL
Injection speed	100 µL/sec
Bubble elim. pull-up cycle	2

Parameter	Set
Bubble elimination delay	15 seconds
Sample wash cycle	1 (or 2)
Depth inside injector	30-35 mm
Needle speed	8

OPERATING SEQUENCE

Performing a S/SL Injection

Use the following sequence to inject a sample into a split/splitless injector.

Before injection, do the following:

- Verify that a column is correctly installed, the correct liner is in the injector, and the system is free of leaks.
- Check the oven temperature and detector temperature.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.
- Verify that you have the proper syringe for the technique you are using:
  - 50 mm needle for split injection
  - 70–75 mm needle for splitless injection

WARNING! Hydrogen is a potentially dangerous gas. Refer to *Using Hydrogen* on page xxviii for safety information.

Manual Injection

1. Press PREP RUN. Depending on the mode you have programmed, the TRACE GC Ultra will perform the following operations:
   - 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.

- In splitless mode, PREP RUN closes the split valve and will close the septum purge valve as programmed.
- In surge splitless mode, PREP RUN initiates the surge pressure.

2. When the **Ready to Inject** LED is lit, insert the syringe into the injector, wait for approximately 2 seconds, inject the sample rapidly, and rapidly remove the syringe from the injector. (This is the *Hot Empty Needle* technique.)
3. Press START.

The GC will complete the analysis as programmed.

Injection Using an AI 3000/AS 3000 Autosampler

Before you begin injection, ensure that you have programmed the autosampler method in the **AUTOSAMPLER** menu and the autosampler sequence in the **SEQUENCE** menu.

1. Press PREP RUN. Depending on the mode you have programmed, the TRACE GC Ultra will perform the following operations:
   - When the gas saver function is programmed, PREP RUN ends the gas saver mode and resets the split flow to the analytical flow.
   - In splitless mode, PREP RUN closes the split valve and will close the septum purge valve as programmed.
   - In surge splitless mode, PREP RUN initiates the surge pressure.
2. Press SEQ CONTROL.
3. Scroll to `Start Sequence` and press ENTER or START.

The autosampler will inject the samples according to the programmed method and sequence.

Automatic Injection Using a TriPlus Autosampler

Refer to the TriPlus Operating Manual and to the manual of the Data System in use.

OPERATING SEQUENCE

Performing a LVSL Injection

Use the following sequence to inject a sample into a large volume splitless injector.

Before injection, do the following:

- Verify that the pre-column and the column are correctly installed, the correct liner is in the injector, and the system is free of leaks.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.
- Verify that you have the proper syringe for the technique you are using:
  - 50 mm needle for LV splitless injection

WARNING! Hydrogen is a potentially dangerous gas. Refer to *Using Hydrogen* on page xxviii for safety information.

Manual Injection

1. Press PREP RUN. Depending on the mode you have programmed, the TRACE GC Ultra will perform the following operations:
   - 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.
   - In splitless mode, PREP RUN closes the split and the septum purge valves as programmed.
2. When the **Ready to Inject** LED is lit, insert the syringe into the injector, inject the sample rapidly, and rapidly remove the syringe from the injector.
3. Press START.

The GC will complete the analysis as programmed.

Automatic Injection Using a TriPlus Autosampler

Refer to the TriPlus Operating Manual and to the manual of the Data System in use.

6

On-Column Injector (OCI)

This chapter describes the On-Column Injector (OCI), on-column injection techniques, and operating sequences.

Chapter at a Glance…

OCI Overview 143
OCI Injection Techniques 148
Manual and Automatic Injections 150
OCI Menu 152

Operating Sequencess

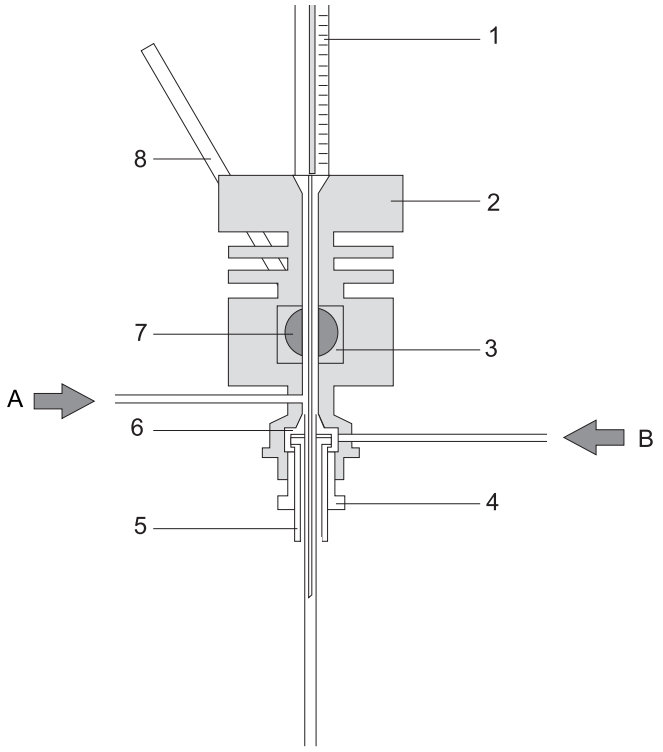
Setting Up the OCI for Manual Injection 153
Programming the OCI 154
Performing an OCI Injection 155

OCI Overview

With on-column injectors, you use a syringe to inject a liquid sample directly into the capillary column.

The upper part of the injector has a needle guide and a rotary valve. The lower part attaches to the top of the column oven. The standard OCI does not have a septum.

The on-column injector is shown in Figure 6-1.



1. Syringe	6. Seal
2. Upper Block	7. Stainless Steel Rotary Valve
3. PTFE Valve Seal	8. Valve Lever
4. M8x1 Fixing Nut	A. Carrier Gas Inlet
5. Cooling Sleeve	B. Secondary Cooling Inlet

Figure 6-1. On-Column Injector

Primary Cooling System

The injection block is kept at ambient temperature by the primary cooling system, which maintains a permanent air flow across the injector body through a special cooling fan.

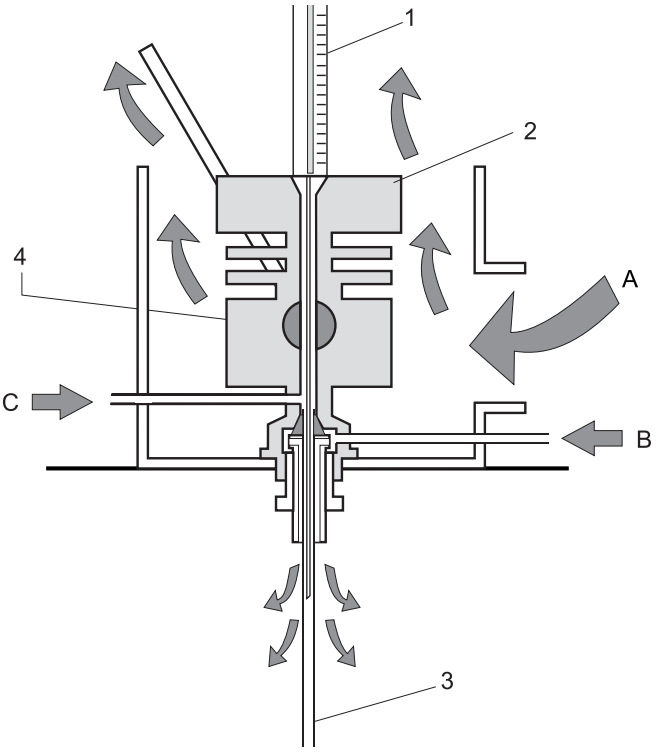
Secondary Cooling System

A gas stream surrounds the area around the column at the injection point. This gas is normally compressed air, but for special applications, CO_2 can be used. The *secondary cooling* flow keeps the injection zone at a temperature below the solvent boiling point, even when the oven runs at a higher temperature. Elevated oven temperature helps eliminate peak distortion in the chromatogram caused by *flooding effects*.[1]

The secondary cooling system ensures complete and effective sample transfer from the syringe to the column and improves reproducibility. Secondary cooling activates immediately before an injection and remains on after the injection until all of the injected solvent has vaporized. The *secondary cooling time*, which is the duration of secondary cooling during a run, depends on the oven temperature, the volatility of the solvent, and the amount injected, but is normally in the range of 3–10 seconds. You program the parameters for secondary cooling in the **`INLET(OCI)`** menu.

1. Journal of Chromatography, 279 (1983) 241–250.

Primary and secondary cooling systems are shown in Figure 6-2.



1. Syringe	A. Primary Cooling
2. Injector Upper Block	B. Secondary Cooling
3. Capillary Column	C. Carrier Gas Inlet
4. Injector Lower Block	

Figure 6-2. Primary and Secondary Cooling Systems

On-Column Options

Optional devices and special on-column injectors can be used for special applications or to help automate certain functions.

Automatic Actuator for Manual Injections

The automatic actuator can semiautomate manual injections by automatically opening the rotary valve when the syringe needle is inserted. When the needle is removed, the automatic actuator closes the valve and starts the GC.

Automatic Actuator for TriPlus Sampler

The automatic actuator for automatic injection with the TriPlus sampler must be installed on the On-Column injector equipped with the appropriate upper block (injection head) to properly open and close the rotary valve of the On-Column injector. The actuator is controlled through its own interface connected to the port marked **DEVICE 1/2** located on the rear portion of the crossrail X of the TriPlus sampler. The automatic actuator for TriPlus is available as kit.

High Oven Temperature (HOT OC) Device

The HOT OC device allows on-column operation at high initial oven temperatures, eliminating the need to cool the oven to a lower temperature for the injection. Chapter 7, *High Oven Temperature Cold On-Column Injector (HOT OC)*, describes this device in detail.

Large Volume On-Column Injector (LVOCI)

The LVOCI is a special version of the standard on-column injector that allows large volume liquid sample analysis with an AS autosampler. Dedicated software is required for this injector. Chapter 8, *Large Volume On-Column Injector (LVOCI)*, describes the principles and hardware for this injection technique.

OCI Injection Techniques

On-column injection is the direct, cold injection of a liquid sample into the column at a point within the column oven and under oven temperature control. The oven temperature determines the actual injection temperature. The injector itself is unheated and serves only as a valve for inserting the syringe needle into the column without depressurizing the column.
The syringe needle enters the injector through a needle channel and passes through a rotary valve and a needle guide. When closed, the rotary valve maintains column pressure. When the valve is open and a syringe needle is inserted, the column pressure remains constant because the needle prevents the gas from escaping.

Cold on-column injection has a number of advantages over the more traditional hot vaporization techniques, from both a qualitative and quantitative viewpoint. Cold injection prevents losses and changes caused by thermal degradation of components in a hot injector. Direct injection without a hot injector vaporization step avoids heavy component discrimination in the syringe needle. When a sample is injected, a plug of liquid forms in the capillary column. This plug of liquid, if uncontrolled, can cause peak distortion. A *flooding effect* occurs when the column's inlet portion floods with liquid sample, up to several meters. You can prevent this effect and maintain perfect peak shapes by carefully controlling the oven temperature during the injection. Oven temperatures of about 10 °C above the solvent boiling point hasten the vaporization of the liquid sample in the column and thus, prevent flooding effects. When using slightly elevated oven temperatures, secondary cooling must be used to control flooding.

Retention Gaps/Pre-Columns

The term *retention gap* refers to an initial part of the column or pre-column that has a much lower retention than the analytical column. A pre-column is a length of fused silica tubing, usually uncoated, connected between the injector and the analytical column. A pre-column protects the analytical column from particulate material (dirt) injected with the sample. A pre-column, when uncoated, can also function as a retention gap.

We recommend using an uncoated length of pre-column in on-column injection for a number of reasons:

- It protects the analytical column from dirt present in the sample. The effect of dirty samples is magnified in on-column injection because the sample is injected directly into the column system.
- It can function as a retention gap. Uncoated retention gaps can tolerate the presence of liquid flooding through them (the flooding effect). Using a retention gap of fused silica limits the flooded zone to a part of the column where chromatography does not take place. Solvent vaporization takes place within the uncoated retention gap so liquid sample does not reach the analytical column. This eliminates peak distortion due to flooding. Injection can take place at oven temperatures below the solvent boiling point, if necessary.
- Wide-bore retention gaps allow fully-automated on-column injection in small diameter capillary columns using an autosampler.

Flooding can also occur during splitless injection, especially with injection volumes greater than 1 µL. The use of retention gaps helps control flooding effects in splitless injection.

For optimal cold on-column injection performance, do not start rapidly programming the oven temperature until the solvent vaporization is complete. The sample is injected with the oven temperature below or, with secondary cooling, moderately above the solvent boiling point using a syringe with a needle made specifically for on-column injection. Refer to Table 6-1.

Table 6-1. On-Column Injection Needles

Needle Type	Application
75 mm length metal needle; 0.23 mm OD	injections into columns with at least 0.3 mm ID
75 mm length fused silica needle; 0.17 mm OD	injection into columns with 0.2–0.25 mm ID
80 mm length metal needle; 0.47 mm OD	automatic standard injections into 0.53 mm OD column with TriPlus autosampler
80 mm length metal needle; 0.23 mm OD	automatic direct injections into 0.25/0.32 mm OD column with TriPlus autosampler

The standard injector upper block (head) has a needle guide with 0.3 mm ID. Fused silica needles require a special upper block with a needle guide of 0.2 mm ID. The automatic injections performed through autosampler require a dedicated injector upper block.

Manual and Automatic Injections

To perform manual or automatic injections, the injector must be equipped with the appropriate upper block (injection heads).

Manual Injections

To perform a manual injection, use a syringe and manually open and close the rotary valve with the valve lever. Refer to the *Setting Up the OCI for Manual Injection* operating sequence on page 153 for instructions.

Automatic Injection with TriPlus Autosampler

You can use the TriPlus autosampler and the dedicated on-column injector head and the automatic actuator to perform automatic injections.

The device allows the opening of the On-column injector rotary valve when the syringe needle is inserted.

When the needle is removed, the actuator closes the valve.
Figure 6-3 shows the automatic actuator for automatic injections with TriPlus autosampler.

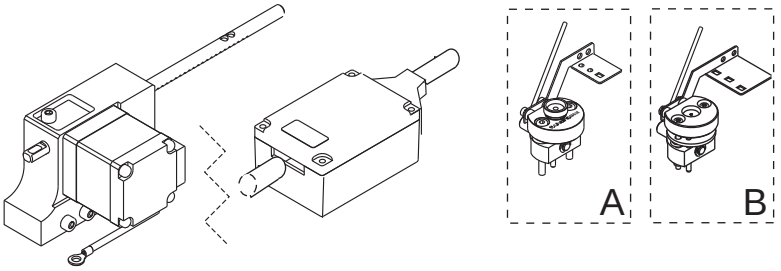


Figure 6-3. Automatic Actuator for Injections with TriPlus Autosampler

- Use the head "**A**" to perform automatic injections into a **0.53 mm ID** capillary column. This head requires the use of an o-ring seal instead of a septum.

- Use the head “**B**” to perform automatic injections into a **0.25/0.32 mm ID** capillary column without the need of using a pre-column. The closure of the vial should be performed by using a tin foil and elastomeric o-ring that guarantees sealing with the standard aluminium cap.

TRACE GC Ultra does not control TriPlus autosampler. The functions of the TriPlus can be controlled through:

- a data processing system for PC with dedicated software.
- a Thermo Scientific Data System.

OCI Menu

The **INLET** menu contains the parameters for on-column injector operations if you have configured an on-column injector.

Press LEFT INLET or RIGHT INLET to display the **INLET (OCI)** menu, depending on the injector position.

NOTE The injector and carrier gas menus are related. If you set a pressure at the carrier gas menu, that same pressure setting is reflected in the injector menu, and vice-versa.

Table 6-2. Inlet (OCI) Menu

Menu	Range	Comments
RIGHT INLET (OCI)		This line is the menu title bar.
Pressure	On/Off, 2–250 kPa or 10–1000 kPa[1]	This line shows the pressure. Press ON to display the actual and setpoint values. Press OFF or 0 to display the actual value and turn off inlet pressure, thereby turning off the flow.
Sec. cool time	0–999.99 min, ∞	This line shows the secondary cooling time, which is the duration of the secondary cooling. If programmed, the valve opens in the **Prep Run** stage.

1. 0.3–36 psi, 0.02–2.5 bar; 0.145–145 psi, 0.1–10.00 bar.

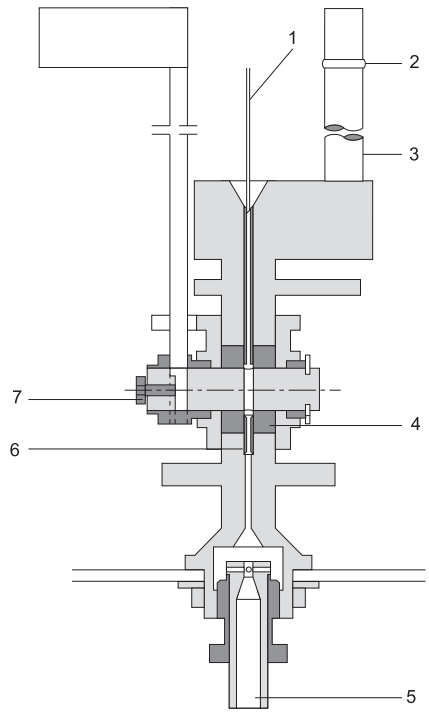
NOTE When you press either COLUMN EVAL or LEAK CHECK while the **INLET** menu is displayed, the GC performs the selected function if the instrument is in the Standby status.

OPERATING SEQUENCE

Setting Up the OCI for Manual Injection

For manual operation, adjust the syringe and needle position in the on-column injector using the needle guide before injecting the sample. See Figure 6-4.

The syringe needle in the injector needle guide prevents possible column depressurization when the valve is open.



1.Syringe needle	5.Secondary Cooling Sleeve
2.Syringe reference	6.Needle Guide
3.Shaft	7.Valve Lever Fixing Screw
4 PTFE Rotary Valve Seal	

Figure 6-4. Manual Injection Setup

1. Close the injection valve and carefully insert the syringe until the needle touches the valve.
2. Withdraw the syringe a few millimeters. This is the correct position for the needle.
3. Adjust and secure the syringe reference accordingly.

CAUTION Check the setting at regular intervals. Failure to do so may result in damage to the syringe needle and to the rotary valve itself if the valve is closed with the syringe needle still in the valve.

OPERATING SEQUENCE

Programming the OCI

Before you begin programming, do the following:

- Verify that a column is correctly installed and the system is free of leaks.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.

1. Press LEFT INLET or RIGHT INLET to open the **INLET** menu, depending on the position of the on-column injector.
2. Scroll to `Sec. cool time` and set the duration of the secondary cooling event.

NOTE The secondary cooling time must be entered in minutes. For example, you would enter `0.10` for a secondary cooling time of 6 seconds.

OPERATING SEQUENCE

Performing an OCI Injection

Use the following sequence to inject a sample into an on-column injector.

Before injection, do the following:

- Verify that a column is correctly installed and the system is free of leaks.
- Check the oven temperature and detector temperature.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.

NOTE

If you do not have an automatic valve actuator installed, you must first perform the *Setting Up the OCI for Manual Injection* operating sequence on page 153.

WARNING!

Hydrogen is a potentially dangerous gas. Refer to *Using Hydrogen* on page xxviii for safety information.

Manual Injection without an Automatic Actuator

1. Press PREP RUN to turn on the secondary cooling flow, if programmed.
2. When the **Ready to Inject** LED is lit, insert the needle of the syringe loaded with sample into the injector needle guide until the barrel of the syringe rests on the preset syringe guide. Refer to the *Setting Up the OCI for Manual Injection* operating sequence on page 153 for information on setting up the syringe guide.
3. Open the valve.
4. Insert the syringe through the valve and into the column as far as it will go.
5. Rapidly inject the sample.
6. Remove the syringe until the syringe barrel rests on the syringe guide.
7. Close the valve.
8. Press START.

9. Remove the syringe completely from the injector.

The GC completes the analysis as programmed.

Manual Injection with an Automatic Actuator

1. Press PREP RUN to switch on the secondary cooling flow, if programmed.
2. When the **Ready to Inject** LED is lit, insert the needle of the syringe loaded with sample through the actuator and into the injector needle guide as far as it will go.
3. Rapidly inject the sample.
4. Rapidly remove the syringe completely from the injector/actuator.

You need not press START. The GC will complete the analysis as programmed.

Automatic Injection Using an AI 3000/AS3000 Autosampler

- Before you begin the autosampler injection, ensure that you have programmed the autosampler method in the **AUTOSAMPLER** menu and the autosampler sequence in the **SEQUENCE** table.

1. Press PREP RUN to turn on the secondary cooling flow, if programmed.
2. Press SEQ CONTROL.
3. Scroll to `Start Sequence` and press ENTER or START.

The autosampler will inject the samples according to the programmed sequence.

Automatic Injection Using a TriPlus Autosampler

Refer to the TriPlus Operating Manual and to the manual of the Data System in use.

7

High Oven Temperature Cold On-Column Injector (HOT OC)

This chapter describes the High Oven Temperature Cold On-Column (HOT OC) injector for injections at very high temperatures, injection techniques, and operating sequences.

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HOT OC Injection Techniques 159
HOT OC Injector Menu 160

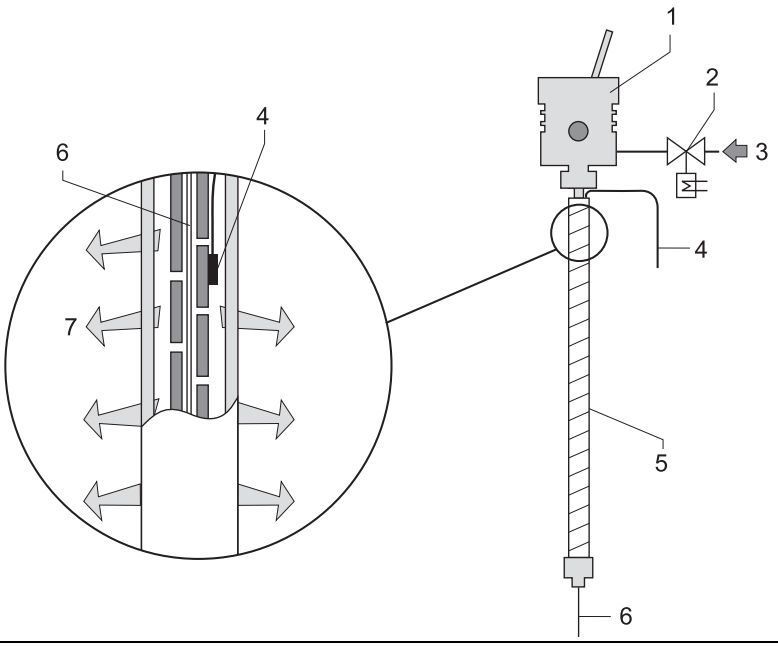
Operating Sequences

Programming the HOT OC Injector 161
Performing a HOT OC Injection 162

HOT OC Overview

The On-Column Injector (OCI) described in Chapter 7 requires an optional device for injection at oven temperatures at or above 200 °C, regardless of the solvent used. A High Oven Temperature (HOT) device must be attached below the on-column injector and configured in the **CONFIGURE** menu.

As with the standard on-column injector, you can manually inject samples into the HOT OC injector with or without an automatic valve actuator. Refer to *On-Column Options* in Chapter 6 for more information about the automatic actuator. Figure 7-1 shows the HOT OC injector.



1. Cold On-Column Injector
2. Solenoid Valve
3. Coolant Inlet
4. Thermocouple
5. HOT Device
6. Capillary Column
7. Coolant Exit

Figure 7-1. HOT Cold On-Column Injector

Optional Devices

In addition to the automatic actuator, the OCI with the HOT device can be modified with a solvent vapor exit valve.

Solvent Vapor Exit Valve

Large volume injection with the HOT OC requires an optional solvent vapor exit (SVE) valve. This valve vents solvent vapors that form during the sample injection. The SVE valve is an electronically activated, heated three-way valve.

The valve inlet connects to a tee piece that links the desolvation pre-column to the analytical column. The solvent vapors vent through the main outlet, which connects to a solvent waste bottle. The SVE valve has a high flow restrictor. This restrictor, a fine capillary tube, is placed in a special support heated by the valve. This configuration ensures a very small purge rate (around 0.01 mL/min) when the SVE valve is closed. This prevents solvent vapor back-diffusion into the analytical system.

HOT OC Injection Techniques

The HOT OC injection technique allows cold on-column injection even when the oven is kept at high temperatures. This can greatly reduce the analysis time.

This technique's advantages are:

- short analysis time, because there is no need to cool to a low oven temperature for injection
- short residence time for components affected by column activity
- isothermal analysis of high boiling components
- reduced effects of column bleed and carrier gas impurities

This technique is limited to a sample size of 1µl or less.

HOT OC Injector Menu

The **INLET (HOT OC)** menu contains the parameters for the HOT OC injector.

Press LEFT INLET or RIGHT INLET to display the menu shown in Table 7-1.

NOTE

The injector and carrier gas menus are related. If you set a pressure at the carrier gas menu, that same pressure setting is reflected in the injector menu, and vice-versa.

Table 7-1. Inlet (HOT OC) Menu

Menu	Range	Comments
RIGHT INLET (HOT OC)		This line is the menu title bar.
HOT OC temp	25 °C–initial oven temp	This parameter defines the injector temperature.
HOT OC duration	0.00–999.99 min, ∞	This parameter defines the duration of the secondary cooling. When programmed, the secondary cooling valve is opened during **Prep Run**. If set to zero, the valve remains in the default condition.
Pressure	On/Off, 2–250 kPa or 10–1000 kPa[1]	This line shows the carrier gas inlet pressure. Press ON to display the actual and setpoint values. Press OFF or 0 to display the actual value and turn off the inlet pressure, which turns off the flow.
SVE temp[2]	On/Off, 0–250 °C	If a solvent vapor exit valve is installed, this parameter defines the SVE valve temperature.
SVE duration[2]	0.00–999.99 min, ∞	This parameter defines the duration of the solvent vapor exit event. When the duration is set to zero, the SVE valve remains in the default condition.

1. 0.3–36 psi, 0.02–2.5 bar; 0.145–145 psi, 0.1–10.00 bar.
2. This menu item appears only if the solvent vapor exit valve option is installed and configured (for large volume injections).

OPERATING SEQUENCE

Programming the HOT OC Injector

Before you begin programming, do the following:

- Verify that the HOT device, together with a column, is correctly installed and the system is free of leaks.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.

1. Press LEFT INLET or RIGHT INLET to open the **`INLET (HOT OC)`** menu, depending on the position of the HOT on-column injector.
2. Scroll to `HOT OC temp` and enter the control temperature for the HOT OC cooling device during injections.
3. Scroll to `HOT OC duration` and enter the time the injector temperature must be maintained. This value depends on the initial injector temperature, solvent boiling point, and sample size.

SVE Valve

1. If the solvent vapor exit valve is installed and configured, scroll to `SVE temp` and enter an appropriate temperature, depending on the solvent boiling point.
2. Scroll to `SVE duration` and enter the time the solvent vapor exit valve must be kept open to allow the solvent to evaporate adequately.

OPERATING SEQUENCE

Performing a HOT OC Injection

Use the following sequence to inject a sample into a cold on-column injector with the HOT device.

Before injection, do the following:

- Verify that the HOT device, together with a column, is correctly installed and the system is free of leaks.
- Check the oven temperature and detector temperature.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.

WARNING! Hydrogen is a potentially dangerous gas. Refer to *Using Hydrogen* on page xxviii for safety information.

Manual Injection without an Automatic Actuator

1. Press PREP RUN. The secondary flow switches on and cools the HOT device to the programmed temperature.
2. When the **Ready to Inject** LED is lit, insert the needle of the syringe loaded with sample into the injector needle guide until the barrel of the syringe rests on the preset syringe guide.
3. Open the valve.
4. Insert the syringe through the valve and into the column as far as it will go.
5. Rapidly inject the sample.
6. Remove the syringe until the syringe barrel rests on the syringe guide.
7. Close the valve.
8. Press START.

9. Remove the syringe completely from the injector. The GC will complete the analysis as programmed.

Manual Injection with an Automatic Actuator

1. Press PREP RUN. The secondary flow switches on and cools the HOT device to the programmed temperature.

2. When the **Ready to Inject** LED is lit, insert the needle of the syringe loaded with sample through the actuator and into the injector needle guide as far as it will go.

3. Rapidly inject the sample.

4. Rapidly remove the syringe completely from the injector/actuator.

You do not need to press START. The GC will complete the analysis as programmed.

Injection Using an AI 3000/AS 3000 Autosampler

Before you begin the autosampler injection, ensure that you have programmed the autosampler method in the **AUTOSAMPLER** menu and the autosampler sequence in the **SEQUENCE** menu.

1. Press PREP RUN. The secondary flow switches on and cools the HOT device to the programmed temperature.

2. Press SEQ CONTROL.

3. Scroll to `Start Sequence` and press ENTER or START.

The autosampler will inject the samples according to the programmed sequence.

Automatic Injection Using a TriPlus Autosampler

Refer to the TriPlus Operating Manual and to the manual of the Data System in use.

8

Large Volume On-Column Injector (LVOCI)

This chapter describes the Large Volume On-Column Injector (LVOCI) used for large volume injections with an autosampler.

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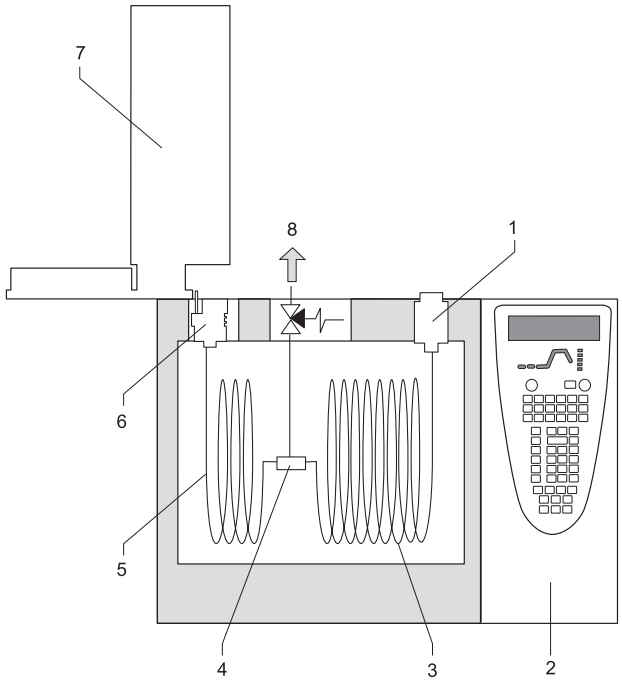
LVOCI Injection Techniques 167
LV On-Column Injector Menu 174

Operating Sequences

Programming the LVOCI 175
Performing an LVOCI Injection 176

LVOCI Overview

The LVOCI is a special version of the standard on-column injector, described in Chapter 7, which automatically introduces large volume liquid samples with the TriPlus or AI/AS 3000 autosampler. The autosampler injects the samples directly into a fused silica capillary column system as shown in Figure 8-1.



1. Detector	5. Desolvation Pre-Column
2. TRACE GC Ultra	6. On-Column Injector
3. Analytical Column	7. Autosampler
4. Tee Connection	8. Solvent Vapor Exit (SVE)

Figure 8-1. Configuration for Large Volume On-Column Injection

The LVOCI system has a Solvent Vapor Exit (SVE) valve, which vents the solvent vapor formed during a sample injection.

The SVE valve is an electronically activated, heated three-way valve. The valve inlet connects to a tee piece linking the desolvation pre-column to the analytical column.

The solvent vapors vent through the main outlet, that connects to a solvent waste bottle with a filter to the atmosphere. The main outlet also connects to a high flow restrictor, which is placed in a special support heated by the valve.

This configuration ensures a very small purge rate (around 0.01 mL/min) when the SVE is closed. This prevents the back-diffusion of solvent inside the system.

LVOCI Injection Techniques

Trace analysis requires injecting relatively large volumes of sample to make better use of the available sample material and to simplify the sample preparation sequence. Figure 8-2 shows the LVOCI injection technique.

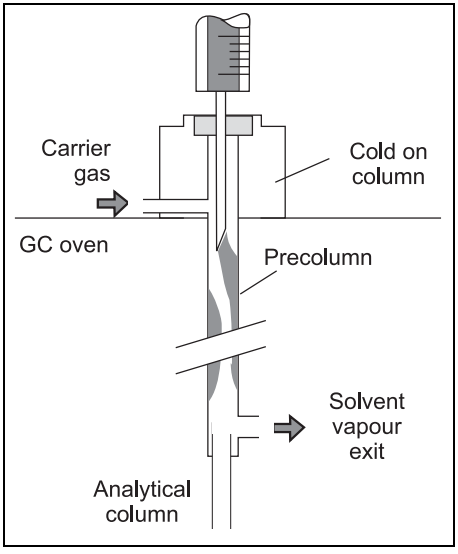


Figure 8-2. LVOCI Injection Technique

Among the available techniques, the on-column injection technique provides the most accurate and reliable results, making it the preferred technique whenever the sample is not excessively dirty.

On-column injection is also the best technique for analyzing volatile components in diluted solutions because of relatively low volatile losses compared to large volume PTV applications.

Mechanism of Sample Desolvation

Figure 8-3 shows the large volume on-column injection system.

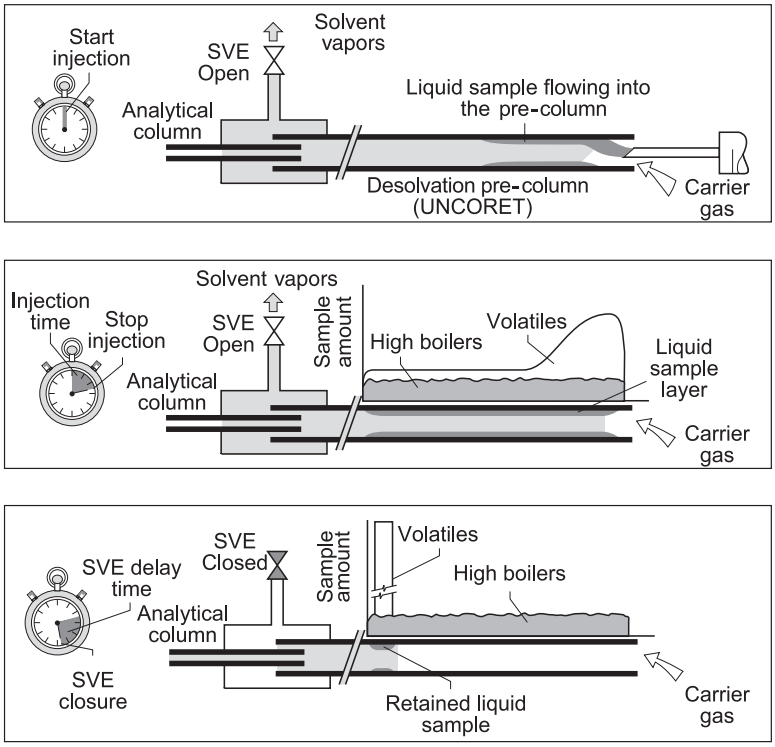


Figure 8-3. Large Volume On-Column Injection System

The liquid sample is injected into a pre-column. The pre-column temperature and pressure conditions cause a part of the solvent to evaporate during injection while the remaining part flows as liquid into the pre-column, forming a flooding zone. This is shown in the top part of Figure 8-3.

The solvent vapors formed during the sample injection are vented through the SVE, located between the pre-column and the analytical capillary column.

Large sample volume on-column injection requires uncoated pre-columns at least as long as the flooded zone. For 0.53 mm ID pre-columns, the zone flooded by 1µL of sample liquid is approximately 10–15 cm long.

Solvent evaporation and solute reconcentration (desolvation) are performed in the 15 m x 0.53 mm ID deactivated (Uncoret™) pre-column which combines the uncoated and the retaining pre-column in one piece. The last 3 m are coated with SE-54/0.45 µm film thickness.

The 12 m x 0.53 mm uncoated section can safely retain about 80 µl of liquid sample. The pre-column ends in a tee union connected to the analytical column and the SVE.

The samples are injected by an autosampler with an adjustable injection speed. Some solvent enters the column, while a large portion of the solvent evaporates and exits through the SVE valve. If the sample volume or the speed of injection exceeds the liquid retention capacity of the uncoated pre-column, the sample enters the column and destroys the chromatography.

An autosampler with adjustable injection speed introduces the sample. Most vapors escape through the open SVE. At the end of the sample injection, the liquid sample coats up to the full length of the uncoated pre-column, as shown in the center part of Figure 8-3.

Solvent evaporation continues, removing solvent from the rear of the sample film. High-boiling components are deposited onto the dry pre-column surface. Volatile components evaporate and are trapped again by the solvent in the pre-column.

Solvent Effects

Solvent effects can be used to trap and reconcentrate samples, increasing the analysis effectiveness.

Solvent Trapping

Liquid sample, advanced by the carrier gas, forms a layer on the column wall. The solvent evaporation proceeds from the rear to the front of this flooded zone, which creates a *solvent trapping effect.* The thick layer of liquid sample retains the volatile components until all solvent evaporates. Thus, all volatile materials start chromatography as a sharp band.

Solvent trapping can be achieved with a small amount of liquid solvent in the pre-column. This allows the impurities from injected solvent to evaporate and vent

through the SVE valve. This is known as *partially concurrent solvent evaporation*.

Phase Soaking

The second solvent effect, *phase soaking*, helps reconcentrate the most volatile sample components not fully trapped or retained by the sample layer. As the carrier gas, saturated with solvent vapor, passes from the sample-coated inlet into the retaining pre-column, the stationary phase film picks up solvent and swells. Depending on the solvent compatibility with the stationary phase, film thickness may increase by a factor of five, which increases retention power. Initial bands are reconcentrated by a dynamic process.

Sample Reconcentration

Liquid sample spreading in the column inlet causes band broadening in space. Components that are not vaporized during the solvent evaporation remain distributed over the whole length of the flooded column inlet. Since initial bands longer than 20–40 cm (sample volumes exceeding 1–2 µL) cause chromatogram peaks to broaden, you must reconcentrate them.

You can reconcentrate the sample by using an uncoated pre-column to achieve a 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.

Retention Gaps

A *retention gap* is the initial part of the column or pre-column with a lower retention power than the analytical column. Retention gaps are recommended for high-resolution capillary gas chromatography for a number of reasons:

- Retention gaps allow you to reconcentrate a broadened inlet band caused by liquid sample flooding eliminating the problem of the flooded zone in splitless and on-column injection.
  The flooded zone is the part of the column that becomes wet with solvent after an on-column or splitless injection because:
  - — liquid sample moves slowly, drastically reducing the analytical column's efficiency due to interference with the chromatographic partition process.
  - — liquid sample interacts with stationary phase. In a flooded zone, the sample solvent can partially strip the stationary phase off the column wall. This can lead to sample contamination and gradual deterioration in the column performance. The sample solvent, if allowed to condense within the analytical column, may even extract a bonded phase, although to a much lesser extent.

  A retention gap of deactivated fused silica limits the flooded zone to a part of the column where no chromatography takes place.
- Retention gaps act as pre-columns for sample containing large amounts of nonvolatile components.
- Wide-bore retention gaps allow fully automated on-column injections in small diameter capillary columns using the autosampler.

Uncoret™ Pre-Columns

The Uncoret™ pre-column is a deactivated fused silica wide-bore column that consists of a 15 m long, 0.53 mm ID, where the first 12 m are uncoated pre-column that functions as a retention gap, and the last 3 m are coated segment (SE-54/0.45 µm film thickness), which functions as a retaining pre-column.

The coated section reduces the eventual loss of volatile substances during the last solvent evaporation phase.

This special pre-column receives the solvent/sample when it is injected with the autosampler syringe at the appropriate speed through the on-column injector.

When the sample is injected inside the empty pre-column, equilibrium is established between the evaporating solvent and the liquid deposited on the precolumn wall. The solvent vapor exit valve speeds up solvent evaporation.

To avoid sample loss, the injected liquid must not exceed the liquid capacity of the uncoated part of the pre-column. This technique prevents liquid sample from entering the stationary phase of the retaining pre-column.

The wet zone length depends on the solvent type, the flow, and the pressure and temperature conditions in the pre-column.

The Uncoret™ pre-column attaches to a 0.32 mm ID or 0.25 mm ID fused silica capillary column with a tee connector.

Early Vapor Exit

The solvent vapors formed by the sample desolvation exit through the *early vapor exit*. The vapor exit is positioned at the earliest possible point to shorten the vapor flow path to a minimum and to achieve a maximum discharge rate at a given inlet pressure.

A maximum split ratio can be achieved at the tee union dividing the flow from the pre-column between the vapor exit and the analytical column. This can minimize the amount of vapor reaching the detector.

A section of coated pre-column retains solutes until the vapor exit closes. The vapor exit is usually closed shortly before the solvent evaporation ends. The residual liquid still retains the volatile components. See the bottom of Figure 8-3 on page 168. Remaining solvent exits through the separation column. When solvent evaporation is completed, volatile components start the chromatography process.

The reconcentration of components with high boiling points occurs as they move to the analytical column entrance at an increased oven temperature.

System Regulation

Partially concurrent solvent evaporation and using an early vapor exit complicate selecting appropriate analytical conditions.
Partially concurrent solvent evaporation requires sample introduction at a rate slightly above the solvent evaporation rate in the pre-column.

- A slower introduction causes all solvent to evaporate concurrently, eliminating solvent trapping.
- A faster introduction rate, however, results in flooding the retaining pre-column and eventually the column, because there is an insufficient proportion of the solvent evaporating concurrently.

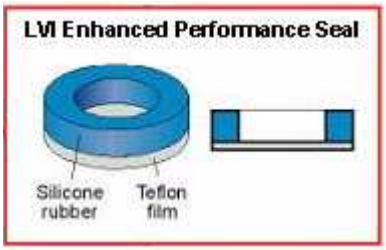
The early vapor exit must be closed as late as possible, after most of the solvent has been evaporated, but before the solute material of interest starts leaving.

With solvent trapping, the sample film retains volatile components up to the end of the solvent evaporation. You can safely close the exit valve shortly before the end of the solvent evaporation.

Control of operations such as evaporation and injection is automatically carried out through the large volume software.

Automatic Injections

The autosampler performs automatic large volume on-column injections. The injector must be equipped with the appropriate upper block for automatic injections.



When performing Large Volume Injections with the autosampler, it is strongly recommended to use LVI enhanced performance vial seals PN 313 010 01 (20 mm) or PN 313 011 02 (11 mm) or PN 313 010 03 (8 mm). These seal will avoid contamination with silicon, common to the conventional vial seals. These seals can be used for no more than 2-3 injections, depending on solvent volatility.

LV On-Column Injector Menu

The **INLET (LVOCI)** menu contains the parameters for large volume on-column injectors if the GC has been configured for an LVOCI.

Press LEFT INLET or RIGHT INLET to display the menu, depending on the injector position.

Table 8-1. Inlet (LVOCI) Menu

Menu	Range	Comments
RIGHT INLET (LVOCI)		This line is the menu title bar.
Pressure	On/Off, 2–250 kPa or 10–1000 kPa[1]	This line shows the carrier gas inlet pressure. Press ON to display the actual and setpoint values. Press OFF or 0 to display the actual value and to turn off the inlet flow.
Sec. cool time	0–999.99 min, ∞	This line shows the secondary cooling time. If set to ∞, the solenoid valve remains in the default condition. The valve opens at the beginning of the **Standby** mode, when programmed.
SVE temp[2]	On/Off, 0–250 °C	This line only appears when the optional solvent vapor exit valve is installed in the system. This parameter defines the solvent vapor exit valve temperature.
SVE duration[2]	0–999.99 min, ∞	This parameter defines the duration of the solvent vapor exit event. When the duration is set to zero, the SVE valve remains in the default condition.
Evap pressure[2]	2–250 kPa or 10-1000 kPa	This parameter defines the pressure used during the solvent evaporation phase.
Evap duration[2]	0-999.9 min	This parameter define the duration of the evaporation event.

1. 0.3–36 psi, 0.02–2.5 bar; 0.145–145 psi, 0.1–10.00 bar.
2. This menu item appears only if the solvent vapor exit valve option is installed and configured.

OPERATING SEQUENCE

Programming the LVOCI

The liquid sample is introduced directly into a pre-column within the column oven. The injector itself is cooled independently. The oven temperature and the secondary cooling system determine the actual injection temperature.

The LVOCI has special PC-based software that calculates all the critical injection parameters for the large volume injection technique.

Before downloading the calculated data to the GC, do the following:

- Verify that an Uncoret™ retaining pre-column and analytical column are correctly connected to the low-volume tee piece and the SVE valve. For instructions on connecting the columns, refer to Chapter 14, *Columns*.
- Verify that the system is free of leaks.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.

Once you have downloaded the injection parameters to the GC, you are ready to begin the injection sequence.

OPERATING SEQUENCE

Performing an LVOCI Injection

Use the following sequence to inject a sample into an LVOCI.

Before injection, do the following:

- Verify that an Uncoret™ retaining pre-column and analytical column are correctly installed to the low-volume tee piece and the SVE valve. For instructions on connecting the columns, refer to Chapter 14, *Columns*.
- Verify that the system is free of leaks.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.

NOTE

Before you begin an autosampler injection, ensure that you have downloaded the large volume injection parameters from the large volume software and programmed the autosampler sequence.

Refer to the TriPlus Operating Manual and to the manual of the Data System in use.

9

Packed Column Injector (PKD)

This chapter describes the Packed (PKD) column injector and explains the packed column operating sequences.

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PKD Injector Menu 180

Operating Sequences

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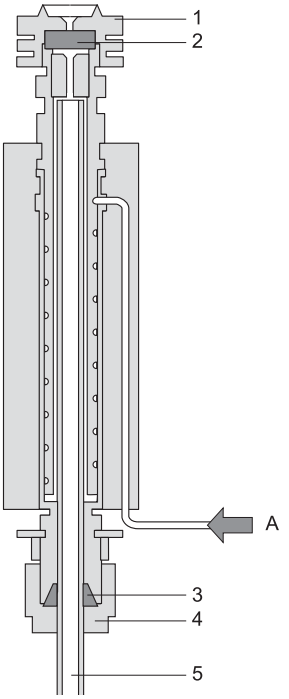
Programming the PKD Injector 182
Performing a PKD Injection 182

PKD Overview

The PKD injector, shown in Figure 9-1, is used for injections with the sample vaporizing directly in the column. The PKD standard injector accepts metal or glass packed columns. The injector temperature may range from ambient to 400 °C. Injector temperature is regulated by a temperature controller in the GC CPU board and monitored by a platinum wire sensor.



1. Septum Cap	4. Column Nut
2. Septum	5. Packed Column
3. Column Ferrule	A. Carrier Gas Inlet

Figure 9-1. Packed Column Injector

Septa

You should use a good quality septum with a long life expectancy, good resistance to deformation, and a low bleed level, even at high temperatures. Additionally, you can use high-temperature septa for both manual and automatic injections.

Adapters

You must install a proper different glass liners depending on the type of column used. Table 9-1 shows the PKD adapter options.

Table 9-1. Adapters for Packed Column Injectors

Adapter	Type of Column
1	packed column 1/4-inch and 6-mm OD
2	packed column 4-mm OD
3	packed column 1/8-inch OD

PKD Injection Techniques

The sample is normally injected directly into the top of the column. The inlet temperature should be sufficiently high to guarantee complete sample vaporization while avoiding the possible decomposition of sample components.

A glass liner prevents nonvolatile substances present in a sample from contaminating the column.

PKD Injector Menu

The **INLET (PKD)** menu contains the parameters for packed columns. Press LEFT INLET or RIGHT INLET to display the menu, depending on the injector position.

NOTE

The injector and carrier gas menus are related. If you set a pressure at the carrier gas menu, that same pressure setting is reflected in the injector menu, and vice-versa.

Table 9-2. Inlet (PKD) Menu

Menu	Range	Comments
XXXX INLET (PKD)		This line is the menu title bar.
Temp	On/Off, 50–400 °C	This line shows the base injector temperature. Press ON to turn on the heater and display the actual and setpoint values. Press OFF to turn off the heater and to display the actual value.
Pressure	On/Off, 2–250 kPa or 10–1000 kPa[1]	This line shows the carrier gas inlet pressure. Press ON to display the actual and setpoint values. Press OFF or 0 to display the actual value and to turn off the inlet flow.

1. 0.3–36 psi, 0.02–2.5 bar; 0.145–145 psi, 0.1–10.00 bar.

OPERATING SEQUENCE

Replacing a Septum

Materials required**:**

- septum
- tweezers

WARNING! The injector fittings may be hot. Make sure the injector is at room temperature before replacing the septum.

1. Remove the septum cap from the injector.
2. Using tweezers, remove the septum from the septum cap.
3. Place a new septum in the septum cap.

CAUTION To avoid contamination, do not touch the septum with your hands.

4. Gently tighten the septum cap onto the injector assembly until finger-tight.

   Do not overtighten the septum cap. The septum will deform and may be difficult to penetrate with the syringe needle.

OPERATING SEQUENCE

Programming the PKD Injector

Before you begin programming, do the following:

- Verify that a column is correctly installed, the correct adapter is in the injector, and the system is free of leaks.
- Check the oven temperature and detector temperature.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.

1. Press LEFT INLET or RIGHT INLET to open the **`INLET (PKD)`** menu, depending on the position of the PKD injector.

2. Scroll to `Temp`, press ON, then enter the appropriate injector temperature using the numeric keypad.

OPERATING SEQUENCE

Performing a PKD Injection

Use the following sequence to inject a sample into a PKD injector.

Before injecting the sample, do the following:

- Verify that a column and adapter, is correctly installed and the system is free of leaks.
- Check the oven temperature and detector temperature.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.

WARNING! Hydrogen is a potentially dangerous gas. Refer to *Using Hydrogen* on page xxviii for safety information.

Manual Injection

1. Press PREP RUN.
2. When the **Ready to Inject** LED is lit, insert the syringe into the injector, inject the sample rapidly, and remove the syringe from the injector.
3. Press START.

The GC will complete the analysis as programmed.

Injection Using an AI 3000/AS 3000 Autosampler

Before you begin the autosampler injection, ensure that you have programmed the autosampler method in the **AUTOSAMPLER** menu and the autosampler sequence in the **SEQUENCE** menu.

1. Press PREP RUN.
2. Press SEQ CONTROL.
3. Scroll to `Start Sequence` and press ENTER or START.

The autosampler will inject the samples according to the programmed sequence.

Automatic Injection Using a TriPlus Autosampler

Refer to the TriPlus Operating Manual and to the manual of the Data System in use.

10

Purged Packed Column Injector (PPKD)

This chapter describes Purged Packed (PPKD) column injector, which has a septum purge. Included in this chapter are PPKD injection techniques and operating sequences.

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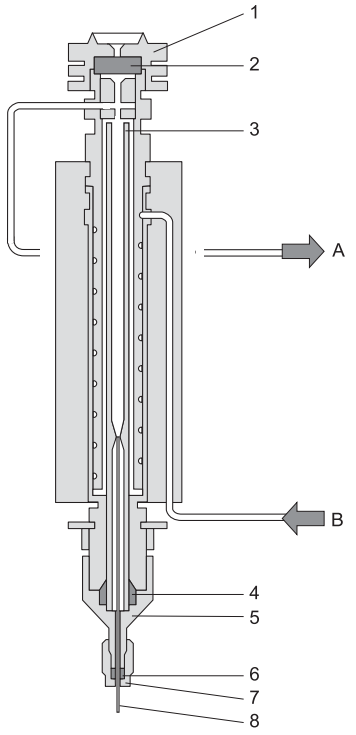
Programming the PPKD Injector Wide-Bore With Surge Mode 192
Programming the PPKD Injector Packed Mode 193
Programming the PPKD Injector Packed With Surge Mode 194
Performing a PPKD Injection 195

PPKD Overview

The Purged Packed (PPKD) column injector is a packed column injector with a septum purge. The PPKD standard injector accepts wide-bore capillary columns. The sample vaporizes in a liner and enters the wide-bore capillary column. The injector temperature is controllable from 50 °C to 400 °C. Figure 10-1 shows the PPKD injector.



1. Septum Cap	6. Column Ferrule
2. Septum	7. M4 Split Retaining Nut
3. Liner	8. Wide-Bore Column
4. Liner Ferrule	A. Septum Purge Line
5. Adapter	B. Carrier Gas Inlet

Figure 10-1. Purged Packed Column Injector

Septa

You should use high temperature septa with a longer life expectancy, good resistance to deformation, and a low bleed level, even at high temperatures. Use high temperature septa for both manual and automatic injections.

Liners

Two different glass liners can be used for wide-bore capillary columns:

- 2 mm ID
- 4 mm ID

PPKD Injection Techniques

The inlet temperature should be sufficiently high to guarantee the sample completely vaporizes while avoiding the possible sample component decomposition.

PPKD Injector Menu

The **INLET (PPKD)** menu contains the operating parameters for the purged packed injector. The parameters you can edit depend on the operating mode chosen: `Wide bore`, `Packed`, `Wide bore w/surge` or `Packed w/surge`.

- In the `Wide bore` and `Wide bore w/surge` operating modes, the column flow is regulated by changing the pressure as the temperature changes.
- In the `Packed` and `Packed w/surge` operating modes, the column flow is controlled through true mass flow control.

Press LEFT INLET or RIGHT INLET to open the **LEFT** or **RIGHT INLET (PPKD)** injector menu.

```
      LEFT INLET (PPKD)

  Temp              250  250
  Pressure       10.6   10.6
  Mode:               Packed<
```

The Mode: menu item displays the current operating mode.

Press MODE/TYPE to open the **INLET MODE** submenu.

```
         XX INLET MODE

* Wide bore                  <
  Packed
  Wide bore w/surge
  Packed w/surge
```

Scroll to the mode you want to use and press ENTER to confirm the selection. An asterisk appears on the left of the operating mode selected.

NOTE The injector and carrier gas menus are related. If you set a pressure in the carrier gas menu, that same pressure setting is reflected in the injector menu, and vice-versa.

Table 10-1. Inlet (PPKD) Menu

Menu	Range	Comments
XXXX INLET (PPKD)		This line is the menu title bar.
Temp	On/Off, 0–400 °C	This line shows the base injector temperature. Press ON to turn on the heater and display the actual and setpoint values. Press OFF to turn off the heater and display the actual value.
Pressure	On/Off, 2–250 kPa or 10–1000 kPa[1]	This line shows the carrier gas inlet pressure. Press ON to display the actual and setpoint values. Press OFF or 0 to turn off the inlet flow.
Mode:		This line displays the currently selected operating mode. Press ENTER to open the **INLET MODE** submenu.
Surge pressure	On/Off, 2–250 kPa or 10–1000 kPa[2]	This line indicates the surge pressure. Only used with **packed w/surge** and **wide bore w/surge** modes.
Surge duration	0–999.9 min, ∞	This line displays the duration of surge pressure after run start.
Const sept purge?	Yes/No	Press YES to activate a constant septum purge to continuously flush the injector with a purge flow of 5 mL/min for helium and nitrogen or 10 mL/min for hydrogen.
Stop purge for	0–999.9 min, ∞	This line appears only when Constant septum purge is set to No.

1. 0.3–36 psi, 0.02–2.5 bar; 0.145–145 psi, 0.1–10.00 bar.

OPERATING SEQUENCE

Replacing a Septum

Materials required**:**

- septum
- tweezers

WARNING! The injector fittings may be hot. Make sure the injector is at room temperature before replacing the septum.

1. Remove the septum cap from the injector.
2. Using tweezers, remove the septum from the septum cap.
3. Place a new septum in the septum cap.

CAUTION To avoid contamination, do not touch the septum with your hands.

4. Gently tighten the septum cap onto the injector assembly until finger-tight.

   Do not overtighten the septum cap. The septum will deform and may be difficult to penetrate with the syringe needle.

OPERATING SEQUENCE

Programming the PPKD Injector Wide-Bore Mode

Before programming the purged packed column injector, do the following:

- Verify that a wide-bore column is correctly installed, the correct liner is in the injector and the system is free of leaks.
- Check the oven temperature and detector temperature.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.

WARNING! Hydrogen is a potentially dangerous gas. Refer to *Using Hydrogen* on page xxviii for safety information.

1. Press LEFT INLET or RIGHT INLET to open the **`INLET (PPKD)`** menu, depending on the position of the PPKD injector.
2. Scroll to `Mode:` and press MODE/TYPE.
3. Scroll to `Wide bore` and press ENTER.
4. Scroll to `Temp` and press ON or enter the appropriate injector temperature using the numeric keypad.
5. If constant septum purge is required, scroll to `Const sept purge?` and press YES. If constant septum purge is not required, press NO and scroll to `Stop purge for` to enter the time the purge flow should be interrupted.

OPERATING SEQUENCE

Programming the PPKD Injector Wide-Bore With Surge Mode

In the `Wide bore w/surge` mode, a carrier gas pressure surge activates during the injection phase for a preset time. This surge accelerates the transfer process of the substances from the injector to the column. The pressure surge starts in the **Prep Run** phase and ends at the end of the programmed `Surge duration`.

Before programming the packed column injector, do the following:

- Verify that a wide-bore column is correctly installed, the correct liner is in the injector, if used, and the system is free of leaks.
- Check the oven temperature and detector temperature.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.

WARNING! Hydrogen is a potentially dangerous gas. Refer to *Using Hydrogen* on page xxviii for safety information.

1. Press LEFT INLET or RIGHT INLET to open the **`INLET (PPKD)`** menu, depending on the position of the PPKD injector.
2. Scroll to `Mode:` and press MODE/TYPE.
3. Scroll to `Wide bore w/surge` and press ENTER.
4. Scroll to `Surge pressure` and enter the value of the pressure surge.
5. Scroll to `Surge duration` and enter the duration of the pressure surge.
6. Scroll to `Temp` and press ON or enter the appropriate injector temperature using the numeric keypad.
7. If constant septum purge is required, scroll to `Const sept purge?` and press YES. If constant septum purge is not required, press NO and scroll to `Stop purge for` to enter the time the purge flow should be interrupted.

OPERATING SEQUENCE

Programming the PPKD Injector Packed Mode

Before programming the purged packed column injector, do the following:

- Verify that a column is correctly installed, the correct liner is in the injector, if used, and the system is free of leaks.
- Check the oven temperature and detector temperature.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.

WARNING! Hydrogen is a potentially dangerous gas. Refer to *Using Hydrogen* on page xxviii for safety information.

1. Press LEFT INLET or RIGHT INLET to open the **`INLET (PPKD)`** menu, depending on the position of the PPKD injector.
2. Scroll to `Mode:` and press MODE/TYPE.
3. Scroll to `Packed` and press ENTER.
4. Scroll to `Temp` and press ON or enter the appropriate injector temperature using the numeric keypad.
5. If constant septum purge is required, scroll to `Const sept purge?` and press YES. If constant septum purge is not required, press NO and scroll to `Stop purge for` to enter the time the purge flow should be interrupted.

OPERATING SEQUENCE

Programming the PPKD Injector Packed With Surge Mode

In the `Packed w/surge` mode, a carrier gas pressure surge activates during the injection phase for a preset time. This surge accelerates the transfer process of the substances from the injector to the column. The pressure surge starts in the **Prep Run** phase and ends at the end of the programmed `Surge duration`.

Before programming the packed column injector, do the following:

- Verify that a column is correctly installed, the correct liner is in the injector, if used, and the system is free of leaks.
- Check the oven temperature and detector temperature.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.

WARNING! Hydrogen is a potentially dangerous gas. Refer to *Using Hydrogen* on page xxviii for safety information.

1. Press LEFT INLET or RIGHT INLET to open the **`INLET (PPKD)`** menu, depending on the position of the PPKD injector.
2. Scroll to `Mode:` and press MODE/TYPE then scroll to `Packed w/surge` and press ENTER.
3. Scroll to `Surge pressure` and enter the value of the pressure surge.
4. Scroll to `Surge duration` and enter the duration of the pressure surge.
5. Scroll to `Temp` and press ON or enter the appropriate injector temperature using the numeric keypad.
6. If constant septum purge is required, scroll to `Const sept purge?` and press YES. If constant septum purge is not required, press NO and scroll to `Stop purge for` to enter the time the purge flow should be interrupted.

OPERATING SEQUENCE

Performing a PPKD Injection

Before injecting the sample, do the following:

- Verify that the column and liner, if used, are correctly installed and the system is free of leaks.
- Check the oven temperature and detector temperature.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.

WARNING! Hydrogen is a potentially dangerous gas. Refer to *Using Hydrogen* on page xxviii for safety information.

Manual Injection

1. Press PREP RUN.
2. When the **Ready to Inject** LED is lit, insert the syringe into the injector, inject the sample rapidly, and remove the syringe from the injector.
3. Press START.

The GC will complete the analysis as programmed.

Injection Using an AI 3000/AS 3000 Autosampler

Before you begin the autosampler injection, ensure that you have programmed the autosampler method in the **AUTOSAMPLER** menu and the autosampler sequence in the **SEQUENCE** menu.

1. Press PREP RUN.
2. Press SEQ CONTROL.
3. Scroll to `Start Sequence` and press ENTER or START.

The autosampler will inject the samples according to the programmed sequence.

Automatic Injection Using a TriPlus Autosampler

Refer to the TriPlus Operating Manual and to the manual of the Data System in use.

11

Programmable Temperature Vaporizing Injector (PTV)

This chapter describes the Programmable Temperature Vaporizing (PTV) injector and contains operating sequences for the different PTV operating modes.

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Operating Sequences

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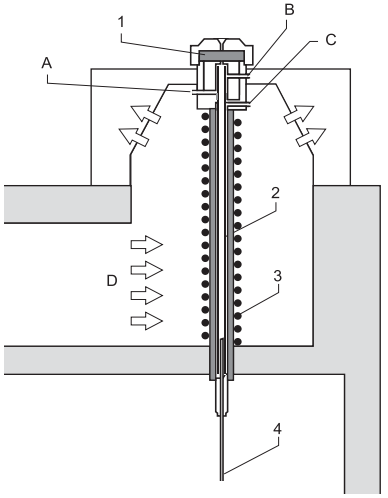
PTV Overview

The BEST (Brightly Enhanced Sample Transfer) PTV injector, shown in Figure 11-1, allows you to vary the temperature during injection in both split and splitless operating modes. This programmable temperature variation can eliminate many of the unwanted effects that can occur with traditional hot injection techniques, such as distillation of the sample within the needle and large vapor clouds inside the injector chamber.



1. Septum	A. Carrier Gas Inlet
2. Liner	B. Septum Purge Line
3. Heating Element	C. Split Line
4. Capillary Column	D. Forced Cooling

Figure 11-1. Programmable Temperature Vaporizing Injector

The BEST PTV can be used in six different operating modes:

- PTV Split, used with concentrated samples when the sensitivity is not a problem.
- PTV Splitless, used for trace analysis.
- PTV Solvent Split, used to vent the solvent or the reagent when it can create a problem for the detector or the column.
- PTV Large Volume, used to increase the sensitivity of the analysis through the injection of large volume sample amount.
- Constant Temp Split, for small sample volume and small volatility range.
- Constant Temp Splitless without or with pressure surge, for small sample volume and trace analysis.

In Constant Temperature (CT) mode, the PTV functions like a split/splitless injector. Sample volumes are lower than when using an S/SL injector because of the smaller PTV liner volume.

The PTV injector can analyze relatively dirty samples that can not be analyzed using a traditional on-column technique.
The injector temperature, from ambient to 400 ºC, is regulated by a temperature controller in the GC CPU card and monitored by a thermocouple.

Liquid nitrogen or liquid carbon dioxide is used as a coolant for operating below ambient temperature (down to -50 with liquid N_2; down to –30 ºC with liquid CO_2). The coolant flow is controlled by an optional cryogenic system which must be connected to the GC and enabled in the **CONFIGURE** menu. Refer to paragraph *PTV Cryogenic Operation* on page 221 and to the *Configuring Cryogenic Operation* on page 239 for more information.

WARNING! Before using liquid nitrogen or liquid carbon dioxide, read the indication of hazard and the instructions reported in the Safety Sheet supplied by the manufacturer with reference to the relevant CAS number (Chemical Abstract Service).

An optional Backflush system is available. Backflushing allows to eliminate during the cleaning phase the heavy part of the sample, which are not relevant for the analysis. It is also able to perform Large Volume Injection reducing the amount of solvent entering the column and increasing the recovery of volatile

components.
Refer to paragraph *PTV Backflush Operation* on page 224 and to the *Enabling Backflush* operating sequence on page 241 for details.

Syringe

A 5-250 µL syringe with a 51 mm, conical-tipped needle or side hole needle for Large Volume injections (with glass sintered liner) are normally used to operate with PTV injector.

Septum

Standard Septum

You should always use good quality septa, such as the BTO septa supplied with the TRACE GC Ultra. Such septa resist deformation, have longer life expectancy, and have a low bleed level, even at high temperatures.

Merlin Microseal™ Valve

PTV injector is compatible with use the Merlin Microseal™ High Pressure Valve instead of the standard septa.

NOTE To replace the standard septum with the Microseal™ Valve, the relevant installation kit is required.

Microseal™ valve requires a 0.63 mm diameter (0.025-inch) blunt tip syringe or the side hole needle tip.

Liners

The selection of the liner depends on the type of application and operating mode needed for your analysis. PTV liners can be glass or silcosteel (stainless steel covered internally with deactivated silica). Figure 11-2 and Table 11-1 shown the PTV liner options.

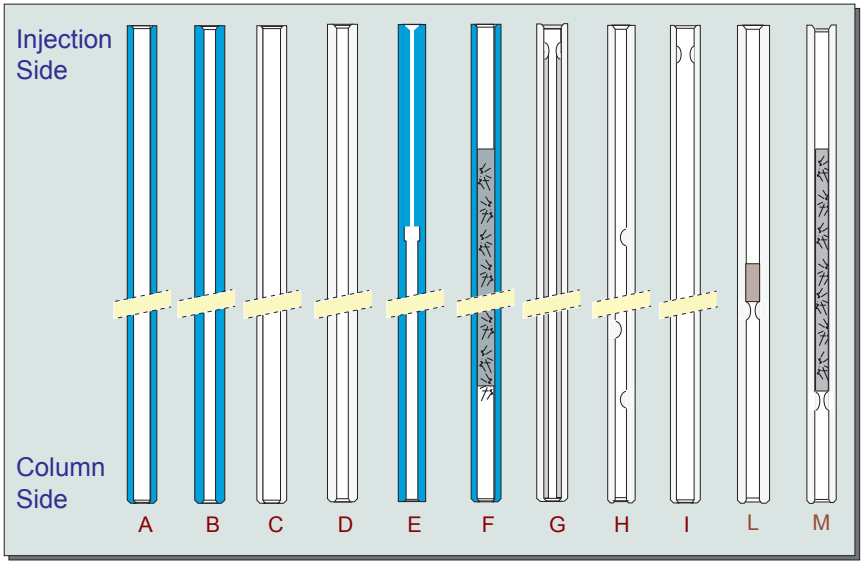


Figure 11-2. PTV Injector Liners

Table 11-1. PTV Injector Liners

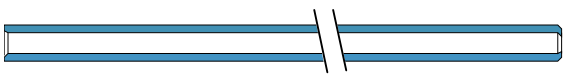
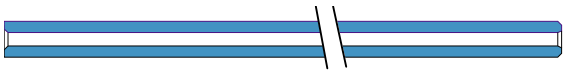
ID#	Part Number	Liner Type Description and Application
A	453 220 44	 2 mm ID; 2.75 mm OD; 120 mm length; 0.38 mL theoretical volume. Silcosteel deactivated liner, used for split and splitless injections. Included in the GC standard outfit as liner for general purpose.
B	453 220 46	 1 mm ID; 2.75 mm OD; 120 mm length; 0.095 mL theoretical volume. Silcosteel deactivated liner, used for splitless injection of samples with high molecular weight compounds. Included in the GC standard outfit standard outfit as liner for general purpose.

Table 11-1. PTV Injector Liners (Continued)

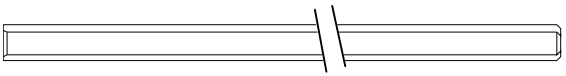
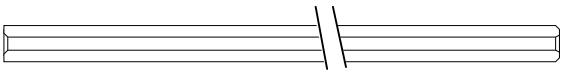
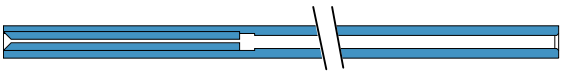
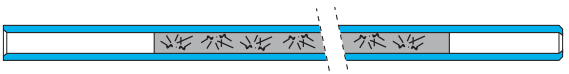
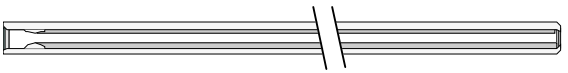
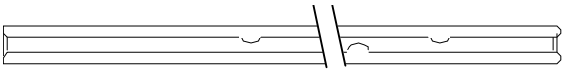
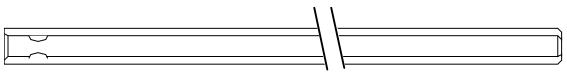
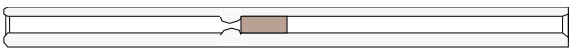
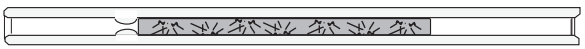
ID#	Part Number	Liner Type Description and Application
C	453 220 45	 2 mm ID; 2.75 mm OD; 120 mm length; 0.38 mL theoretical volume. Non deactivated glass liner, used for split and splitless injections.
D	453 220 54	 1 mm ID; 2.75 mm OD; 120 mm length; 0.095 mL theoretical volume. Deactivated glass liner, used for splitless injection of samples with high molecular weight compounds.
E	453 220 52	 1 mm ID; 2.75 mm OD; 120 mm length; not remarkable theoretical volume. Silcosteel deactivated liner with a 0.6 mm ID restrictor, used when the PTV operates like an on-column injector (refer to *PTV On-Column Like Injection* on page 204). Requires the use of a Wide-bore column or a Wide-bore precolumn.
F	453 220 56	 2 mm ID; 2.75 mm OD; 120 mm length; 0.38 mL theoretical volume. Silcosteel deactivated liner with deactivated silica wool for PTV Large Volume Injections. It is included in the PTVLV standard outfit.
G	453 220 60	 About 1.2 mm ID; 2.75 mm OD; 120 mm length; 0.135 mL theoretical volume.Glass sintered deactivated liner (without quartz wool) for PTV Large Volume Injections with backflush of polar and labile compounds. Used instead of liner **F**. The sintered porous surface coating (0.25-0.5 mm) holds the liquid during the controlled speed LV injection while offering a chemically inert surface.

Table 11-1. PTV Injector Liners (Continued)

ID#	Part Number	Liner Type Description and Application
H	453 220 62	 1 mm ID; 2.75 mm OD; 120 mm length; 0.180 mL theoretical volume. Deactivated glass liner with baffles. The deactivated surface with baffles allows to increase the injectable volumes with the 1 mm ID glass liner. Baffles can also be used for holding small amount of silica wool.
I	453 220 57	 2 mm ID; 2.75 mm OD; 120 mm length; 0.340 mL theoretical volume. Deactivated glass liner: The deactivated glass surface offers the advantage of a chemically inert environment for split and splitless injections of polar compounds.
L	455 220 70	 2 mm ID; 2.75 mm OD; 120 mm length; with silica wool. Deactivated liner with a plug of silica wool. Very suitable for PTV Split operation with Ultrafast GC and for PTVBKF option.
M	453 520 99	 2 mm ID; 2.75 mm OD; 120 mm length. Glass deactivated liner with 60 mm of deactivated silica wool for PTV Large Volume Injections. Used instead of liner **F**.

PTV Injection Techniques

In programmed temperature mode, the sample is injected into the liner in cold conditions. It is rapidly heated to the programmed vaporizing temperature and transferred into the capillary column.
The syringe needle is never significantly heated because the initial temperature of the injector is kept enough low to prevent the sample vaporization inside the needle. Cold injection prevents the discrimination of substances with high boiling points induced by evaporation inside the hot needle.

After injection, the sample vaporizes gradually. This prevents the steam cloud phenomenon common to hot split/splitless injectors. If large enough to exceed the liner volume, a steam cloud escapes through the septum purge line and the split gas line. This phenomenon can also occur if sample volumes are too large or if the initial injection temperature is set too high.

NOTE

The transfer temperature must be kept for the whole analysis time to allow the transfer of heavier components, unless an higher temperature is selected in the Cleaning Phase.

PTV On-Column Like Injection

The PTV injector can be used similarly to an on-column injector if equipped with a special liner which has a restrictor on top. Refer to Table 11-1. The restrictor 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. After a short injection time (5–20 seconds), the injector heats with a programmed rate to reach the sample transfer temperature. When using this technique, set the oven temperature below the solvent boiling point. Set the initial oven time to a value higher than the injection time and the PTV transfer time. You should choose the split mode and select the lowest possible split flow (10–15 mL/min) when using the PTV for this type of injection. Maintain the final PTV temperature up to the end of GC run.

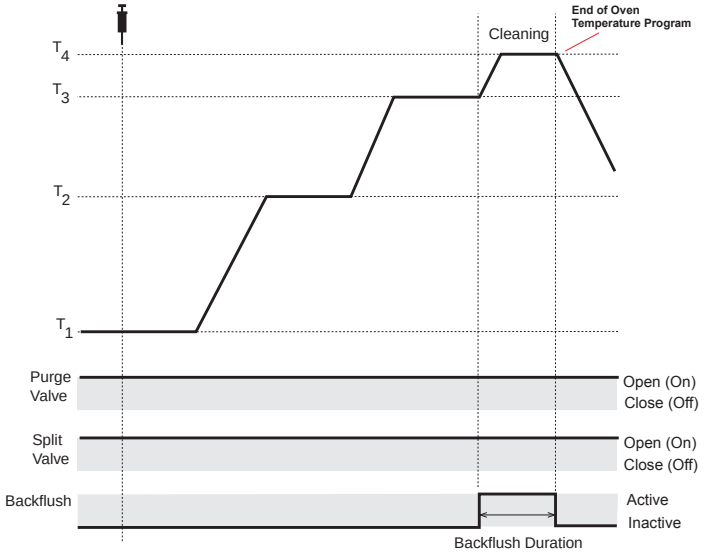
PTV Split Injection

During split injection the splitting valve is open. Only a portion of the sample enters the column. The remainder discharges through the splitting line.
The ratio between the split flow and the column flow defines the amount of

sample that enters the chromatographic system. The split flow must be set to obtain the correct split ratio for the sample concentration to be injected.

The initial temperature should be lower than the solvent boiling point. The final temperature should be suitable for vaporizing the component with the highest boiling point.

An example of temperature profile and timing of the valves in PTV Split mode is shown in Figure 11-3.



T1. Injection Temperature
T3.Sample Transfer Temperature
T2. Evaporation Temperature
T4. Cleaning Temperature

Figure 11-3. Temperature Profile and Timing in PTV Split Mode

PTV Splitless Injection

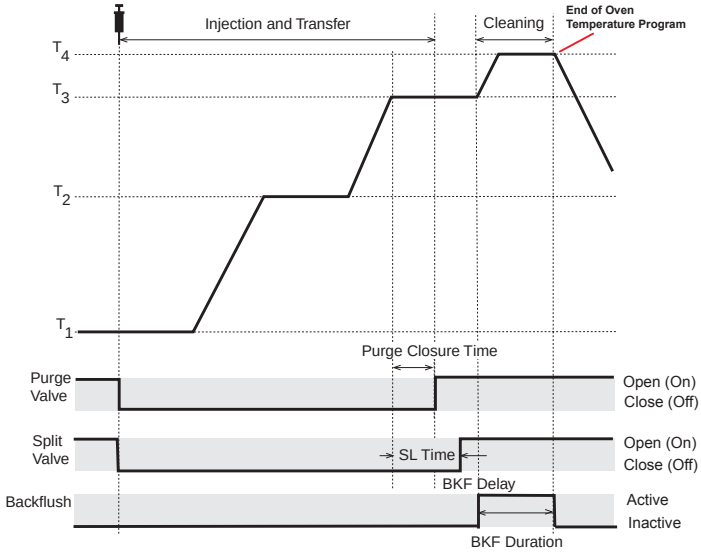
The splitless injection is used primarily to analyze compounds present in very low concentrations, especially in complex matrices. In splitless injection, the splitting valve remains closed during sample injection and transfer into the column.

The time during which the splitting valve remains closed is the *splitless time*. At the end of the sample transfer, the splitting valve opens again to purge the vaporization chamber of residual components, primarily solvent.

The splitless time controls the amount of sample entering the column. This time must end approximately 30–60 seconds after the injector has reached the final temperature. A constant septum purge can continuously flush the injector with a set purge flow throughout the analysis.

The low carrier gas flow during splitless injection causes sampling vapours to fill the vaporization chamber. To avoid sample loss, select an adequate liner, proper temperature conditions, and proper flow conditions.

An example of temperature profile and timing of the valves in PTV Splitless mode is shown in Figure 11-4.



T1. Injection Temperature
T2. Evaporation Temperature
T3.Sample Transfer Temperature
T4. Cleaning Temperature

Figure 11-4. Temperature Profile and Timing in PTV Splitless Mode

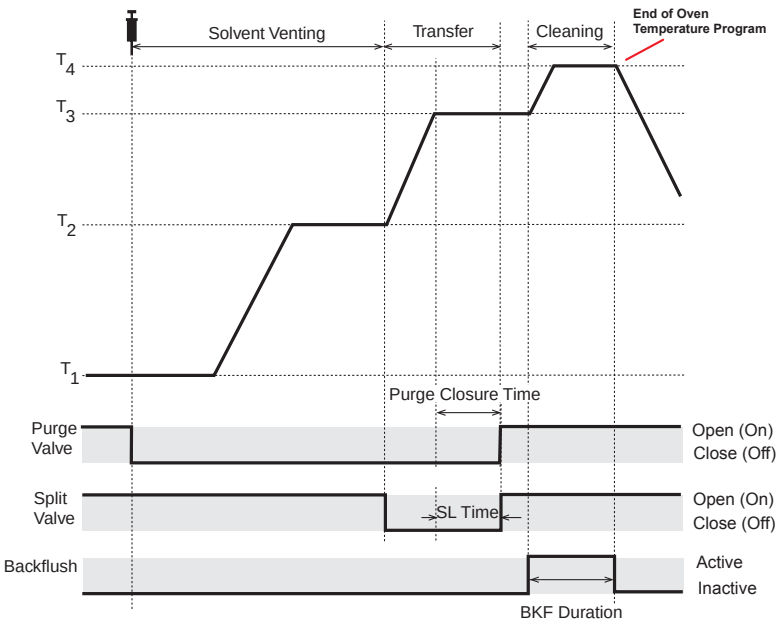
PTV Solvent Split and Large Volume Injections

When your GC is configured with a solvent valve, the PTV Solvent Split operating mode will be replaced with the PTV Large Volume operating mode. For details refer to *PTV Injector Menus* on page 211.

PTV Solvent Split Injection

This technique eliminates the solvent before the sample enters the column. It is used mainly for normal injection volumes if the solvent or derivatizing reagents must be vented.

An example of temperature profile and timing of the valves in PTV Solvent Split mode is shown in Figure 11-5.



T1. Injection Temperature	T3.Sample Transfer Temperature
T2. Evaporation Temperature	T4. Cleaning Temperature

Figure 11-5. Temperature Profile and Timing in PTV Solvent Split Mode

PTV Large Volume Injection

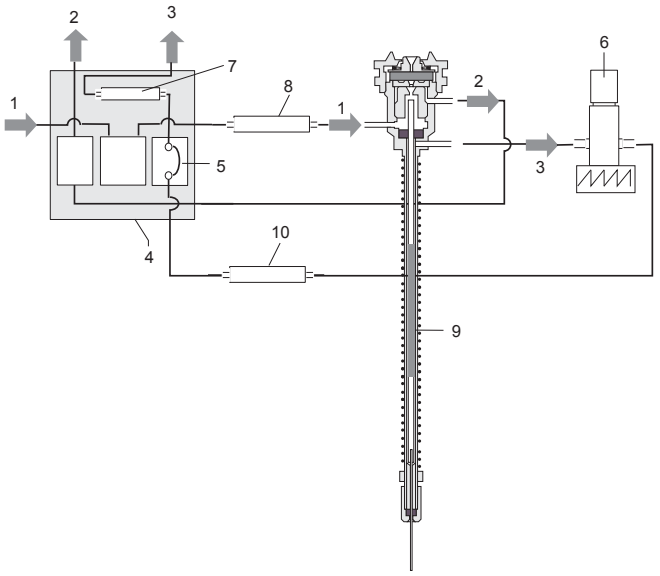
PTV Large Volume injections (PTVLVI) allow large volume injections when the sample components are less volatile than the solvent. In order to operate in the PTVLVI mode, the injector must have a heated solvent split valve installed and configured and adequate DCC configuration.

CAUTION When the heated solvent split valve is installed, the split valve on the DCC module is replaced by the split valve bypass.

Large Volume requires the use of a liner of 2-mm ID with silica wool or other packing material to retain the solvent during injection. The liner is provided in the PTVLVI kit. Alternatively, a sintered glass liner can be used. If your GC has been configured with a solvent valve, the **`INLET (PTV)`** menu contains the parameters for large volume injection.

The PTV injector for Large Volume injections is schematically represented in Figure 11-6.

The Backflush Kit is installed when LVI with backflush is required (less solvent entering the detector and better recovery of volatiles).
Specially designed kit for BKF LVI is available with PN 190 502 38.



1. Carrier Gas Inlet	6. Heated Solvent Split Valve
2. Septum Purge Line	7. Internal Split Line Filter
3. Split Line	8. Carrier Filter
4. DCC Module	9. Liner with silica wool
5. Split Valve Bypass	10. External Split Line Filter

Figure 11-6. PTV Injector for Large Volume Injections

For further details refer to *Large Volume Injections Using PTV* on page 226.

CT Split Injection

This mode is used to execute split injections at a constant temperature. The split and purge valves remain open throughout the run.
Figure 11-7 shows the temperature profile and the timing of the valves.

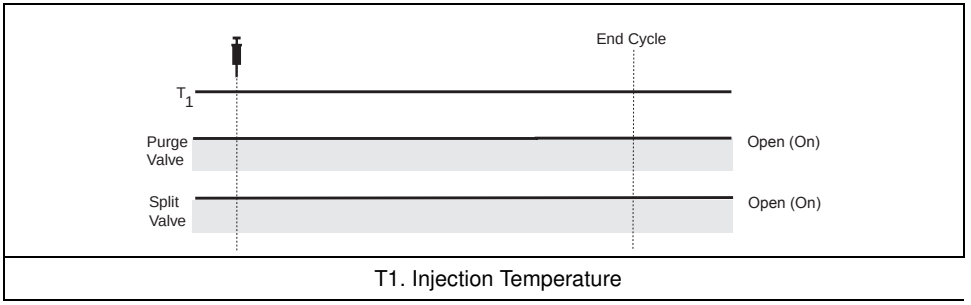


Figure 11-7. Timings of the Valves in CT Split Mode

Sample with limited volatility range is normally injected with this mode.

CT Splitless Injection

This mode is use to execute splitless injections at a constant temperature.
The split and purge valves are closed during the **Prep Run** phase and remain closed after the injection for the programmed duration.
Figure 11-8 shows the temperature profile and the timing of the valves.

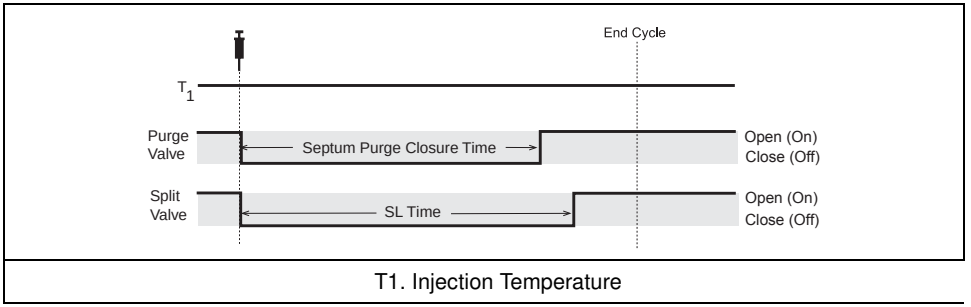


Figure 11-8. Timings of the Valves in CT Splitless Mode

This mode is used for trace analysis of samples with a limited volatility range.

CT Surge Splitless Injection

In CT surge splitless mode, a carrier gas pressure surge activates during the injection phase for a programmed time. This 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.

PTV Injector Menus

The **INLET (PTV)** menu includes the operating parameters for the programmed temperature vaporizing injector. The parameters you can edit depend on the operating mode and temperature mode chosen, and on the type of gas control modules installed in your GC.

- There are three programmed temperature operating modes: PTV split, PTV splitless, and PTV solvent split.
  - — In the programmed temperature modes, you can program the injector temperature to change during an injection. The value you set in the `Temp` parameter acts as a standby temperature.
  - — If your GC has been configured for a solvent valve, the PTV solvent split operating mode will be replaced with the PTV large volume operating mode.
  - — It is possible to program different venting or cleaning flow values during the different phases.
- There are three constant temperature (CT) operating modes:
  - — CT split
  - — CT splitless
  - — CT splitless with surge.

  In the constant temperature modes, the injector operates at the temperature set in `Temp` throughout the analytical run.

Press RIGHT INLET to display the **RIGHT INLET (PTV)** menu. The PTV injector is usually on the right.

```
    RIGHT INLET (PTV)
 Temp              250  250
 Pressure        10.6   10.6
 Mode:                split<
```

The Mode: menu item displays the current operating mode. Press MODE/TYPE to open the **INLET MODE** submenu.

```
     RIGHT INLET MODE
* PTV split             <
  PTV splitless
  PTV solvent split
  CT split
  CT splitless
  CT splitless w/srg
```

NOTE If your GC has been configured for a solvent valve, the PTV solvent split operating mode will be replaced with the PTV large volume operating mode.

Scroll to the mode you want to use and press ENTER to confirm the selection. An asterisk appears beside the selected operating mode. Tables 11-2 through 11-7 explain the ranges and functions of the parameters in the **RIGHT INLET** menus for each operating mode. The items in the inlet menus vary depending on the operating mode you select.

Table 11-2. Inlet (PTV) Menu for Split Mode in Programmed and Constant Temperature

Menu	Range	Comments
RIGHT INLET (PTV)		This line is the menu title bar.
Temp	0–400 ºC, –50–400 ºC with cryo enabled	This line shows the base injector temperature. Press ON to turn on the heater and display the actual and setpoint values. Press OFF to turn off the heater.

Table 11-2. Inlet (PTV) Menu for Split Mode in Programmed and Constant Temperature (Continued)

Menu	Range	Comments
Pressure	On/Off, 2–250 kPa or 10–1000 kPa[1]	This line shows the pressure. Press ON turn on the flow and to display the actual and setpoint values. Press OFF or 0 to turn off the inlet flows and display the actual value.
Mode		This parameter displays the injection operating mode selected. Press ENTER to open the **INLET MODE** selection menu.
Total flow	Not editable	This line shows the total gas flow consumption, which equals the sum of the column flow, split flow (or gas saver flow), and septum purge flow. This value is not editable.
Split flow	On/Off, 0, 10–500 mL/min	This line shows the split flow. Press ON to turn on the split flow and display the actual and setpoint values. Press OFF or 0 to turn off the split flow.
Split ratio	1–5000	This line displays the actual split ratio value, which is the ratio between the split flow and the column flow.
Inject phase menu	Refer to Table 11-7.	Press MODE/TYPE to enter the **INJECT PHASE MENU**. This line appears only in a programmed temperature mode.

1. 0.3–36 psi, 0.02–2.5 bar; 0.145–145 psi, 0.1–10.00 bar.

Table 11-3. Inlet (PTV) Menu for Splitless Mode in Constant and Programmed Temperature

Menu	Range	Comments
RIGHT INLET (PTV)		This line is the menu title bar.
Temp	On/Off, 0–400 ºC, –50–400 ºC with cryo enabled	This line shows the base injector temperature. Press ON to turn on the heater and display the actual and setpoint values. Press OFF to turn off the heater and display the actual value.

Table 11-3. Inlet (PTV) Menu for Splitless Mode in Constant and Programmed Temperature (Continued)

Menu	Range	Comments
`Pressure`	On/Off, 2–250 kPa or 10–1000 kPa[1]	This line shows the pressure. Press ON to turn on the flow and display the actual and setpoint values. Press OFF or 0, to turn off the inlet flows and display the actual value.
`Mode:`		This line displays the selected operating mode.
`Total flow`	Not editable	This line shows the total gas flow consumption, which equals the sum of the column flow, split flow (or gas saver flow), and septum purge flow.
`Split flow`	On/Off, 0, 10–500 mL/min	This line shows the split flow. Press ON to turn on the split flow and turn on the actual and setpoint values. Press OFF or 0 to turn off the split flow.
`Splitless time`	0–999.99 min	This line shows the splitless time, which is the duration of split valve closure.
`Const sept purge?`	Yes/No	This line shows the constant septum purge flow. Press YES to activate the constant septum purge and continuously flush the septum with a fixed purge flow of 5 mL/min for helium and nitrogen or 10 mL/min for hydrogen.
`Stop purge for?`	0–999.99 min, ∞	This line appears only when `Constant sept purge?` is set to `No`.
`Inject phase menu`	Refer to Table 11-7.	Press MODE/TYPE to enter the **`INJECT PHASE MENU`**. This line appears only in a programmed temperature mode.

1. 0.3–36 psi, 0.02–2.5 bar; 0.145–145 psi, 0.1–10.00 bar.

Table 11-4. Inlet (PTV) Menu for Splitless with Surge in Constant Temperature Mode

Menu	Range	Comments
RIGHT INLET (PTV)		This line is the menu title bar.
Temp	0–400 ºC, –50–400 ºC with cryo enabled	This line shows the base injector temperature. Press ON to turn on the heater and display the actual and setpoint values. Press OFF to turn off the heater and display the actual value.
Pressure	On/Off, 2–250 kPa or 10–1000 kPa[1]	This line shows the pressure. Press ON to turn on the gas flow and display the actual and setpoint values. Press OFF or 0, to turn off the inlet flows and display the actual value.
Mode:		This line displays the selected operating mode.
Total flow	Not editable	This line shows the total gas flow consumption, which equals the sum of the column flow, split flow (or gas saver flow), and septum purge flow.
Split flow	On/Off, 0, 10–500 mL/min	This line shows the split flow. Press ON to turn on the split flow and display the actual and setpoint values. Press OFF or 0 to turn off the split flow.
Splitless time	0–999.99 min	This line shows the splitless time, which is the duration of split valve closure.
Surge pressure	2–250 kPa or 10–1000 kPa[1]	This line allows you to program the surge pressure.
Surge duration	0–999.99 min	This line displays the duration of the surge pressure after run start.
Const sept purge?	Yes/No	This line shows the constant septum purge flow. Press YES to activate the constant septum purge and continuously flush the septum with a fixed purge flow of 5 mL/min for helium and nitrogen or 10 mL/min for hydrogen.
Stop purge for?	0–999.99 min, ∞	This line appears only when Constant sept purge? is set to No.

Table 11-4. Inlet (PTV) Menu for Splitless with Surge in Constant Temperature Mode (Continued)

Menu	Range	Comments
Inject phase menu	Refer to Table 11-7.	Press MODE/TYPE to enter the **INJECT PHASE MENU**. This line appears only in a programmed temperature mode.

1. 0.3–36 psi, 0.02–2.5 bar; 0.145–145 psi, 0.1–10.00 bar.

Table 11-5. Inlet (PTV) Menu for Solvent Split Mode

Menu	Range	Comments
RIGHT INLET (PTV)		This line is the menu title bar.
Temp	0–400 ºC, –50–400 ºC with cryo enabled	This line shows the base injector temperature. Press ON to turn on the heater and display the actual and setpoint values. Press OFF to turn off the heater and display the actual value.
Pressure	On/Off, 2–250 kPa or 10–1000 kPa[1]	This line shows the pressure. Press ON to turn on the gas flow and display the actual and setpoint values. Press OFF or 0 to turn off the inlet flows and display the actual value.
Mode		This line displays the selected operating mode.
Total flow	Not editable	This line shows the total gas flow consumption, which equals the sum of the column flow, split flow (or gas saver flow), and septum purge flow.
Split flow	On/Off, 0, 10–500 mL/min	This line shows the split flow. Press ON to turn on the split flow and display the actual and setpoint values. Press OFF or 0 to turn off the split flow.
Splitless time	0–999.99 min	This line shows the splitless time, which is the duration of split valve closure.
Const sept purge?	Yes/No	This line shows the constant septum purge flow. Press YES to activate the constant septum purge and continuously flush the septum with a fixed purge flow of 5 mL/min for helium and nitrogen or 10 mL/min for hydrogen.

Table 11-5. Inlet (PTV) Menu for Solvent Split Mode (Continued)

Menu	Range	Comments
`Stop purge for?`	0–999.99 min, ∞	This line appears only when `Constant sept purge?` is set to `No`.
`Inject phase menu`	Refer to Table 11-7.	Press MODE/TYPE to enter the **`INJECT PHASE MENU`**.

1. 0.3–36 psi, 0.02–2.5 bar; 0.145–145 psi, 0.1–10.00 bar.

In order to operate in large volume mode the PTV must have a solvent valve. All operating mode menus will contain the `Solvent vlv` temperature parameter if a solvent valve has been installed and configured. In any mode the valve must be kept at a minimum temperature of 100 °C.
Table 11-6 shows a typical PTV Large Volume mode menu.

Table 11-6. Inlet (PTV) Menu for Large Volume Mode

Menu	Range	Comments
`RIGHT INLET (PTV)`		This line is the menu title bar.
`Temp`	0–400 ºC, –50–400 ºC with cryo enabled	This line shows the base injector temperature. Press ON to turn on the heater and display the actual and setpoint values. Press OFF to turn off the heater and display the actual value.
`Pressure`	On/Off, 2–250 kPa or 10–1000 kPa[1]	This line shows the pressure. Press ON to turn on the inlet flows and display the actual and setpoint values. Press OFF or 0 to turn off the inlet flows and display the actual value.
`Mode:`		This line displays the selected operating mode.
`Total flow`	Not editable	This line shows the total gas flow consumption, which equals the sum of the column flow, split flow (or gas saver flow), and septum purge flow.
`Split flow`	On/Off, 0, 10–500 mL/min	This line shows the split flow. Press ON to turn on the split flow and display the actual and setpoint values. Press OFF or 0 to turn off the split flow.
`Splitless time`	0–999.99 min	This line shows the splitless time, which is the duration of split valve closure.

Table 11-6. Inlet (PTV) Menu for Large Volume Mode (Continued)

Menu	Range	Comments
`Solvent vlv`	0–160 °C	This line displays the solvent valve temperature.
`Const sept purge?`	Yes/No	This line shows the constant septum purge flow. Press YES to activate the constant septum purge and continuously flush the septum with a fixed purge flow of 5 mL/min for helium and nitrogen or 10 mL/min for hydrogen.
`Stop purge for?`	0–999.99 min, ∞	This line appears only when `Constant sept purge?` is set to `No`.
`Inject phase menu`	Refer to Table 11-7.	Press MODE/TYPE to enter the `Inject Phase` menu.

1. 0.3–36 psi, 0.02–2.5 bar; 0.145–145 psi, 0.1–10.00 bar.

Table 11-7. Inject Phase Menu for Split, Splitless, Solvent Split, and Large Volume Modes

Menu	Range	Comments
`INJECT PHASE`		This line is the menu title bar.
`Ramped pressure?`[5]	Yes/No	Press YES to open the pressure ramp parameters.
`Inject pres`[5]	On/Off, 2–250 kPa or 10–1000 kPa[1]	This parameter defines the pressure value during the injection phase.
`Inject temp`	0–400 °C, –50–400 °C with cryo enabled	This parameter defines the injector temperature during injection.
`Inject time`	0.00–999.99 min	This parameter defines the time to maintain the temperature during and after the injection.
`Vent flow`	10–500 mL/min	This line shows the vent flow during the injection and evaporation phases. It discharges the solvent or the non-retained compounds during the large volume or solvent split phase. The vent flow setpoint must be compatible with the available pressure set.

Table 11-7. Inject Phase Menu for Split, Splitless, Solvent Split, and Large Volume Modes (Continued)

Menu	Range	Comments
Evap pres[2-5]	On/Off, 2–250 kPa or 10–1000 kPa[1]	This parameter defines the pressure used during the solvent evaporation phase. The pressure is applied at the beginning of the evaporation temperature ramp.
Evap ramp[2]	0.1–14.5 ºC/s in 0.1 ºC/s increments	This parameter defines the ramp rate to reach the programmed solvent evaporation temperature.
Evap temp[2]	0–400 ºC, –50–400 ºC with cryo enabled	This parameter defines the solvent evaporation temperature.
Evap time[2]	0.00–999.99 min	This parameter defines the time the programmed solvent evaporation temperature must be maintained.
Transfer pres[5]	On/Off, 2–250 kPa or 10–1000 kPa[1]	This parameter defines the pressure used during the sample transfer phase. This pressure is applied at the beginning of the transfer temperature ramp.
Transfer ramp	0.1–14.5 ºC/s in 0.1 ºC/s increments	This parameter defines the rate of the temperature ramp to reach the sample transfer temperature.
Transfer temp	0–400 ºC, –50–400 ºC with cryo enabled	This parameter defines the temperature at which the sample transfers into the column.
Transfer time	0.00–999.99	This parameter defines the time the programmed sample transfer temperature must be maintained.
Clean ramp[3]	0.1–14.5 ºC/s in 0.1 ºC/s increments	This parameter defines the ramp rate to reach the programmed injector cleaning temperature.
Clean temp[3]	0–400 ºC, –50–400 ºC with cryo enabled	This parameter defines the injector temperature during the cleaning phase.

Table 11-7. Inject Phase Menu for Split, Splitless, Solvent Split, and Large Volume Modes (Continued)

Menu	Range	Comments
`Clean time`[3]	0.00–999.99 min	This parameter defines the time the programmed sample transfer temperature must be maintained.
`Clean flow`[4]	10–500 mL/min	This parameter may be used to increase the flow during the cleaning phase. The clean flow setpoint must be compatible with the pressure set.

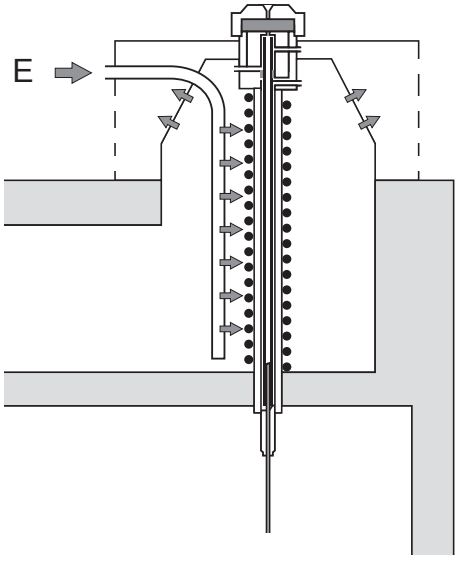
1. 0.3–36 psi, 0.02–2.5 bar; 0.145–145 psi, 0.1–10 .00 bar.
2. This parameter appears only when the `Evaporation?` option has been configured in the **`PTV PHASE EVENTS`** menu. Refer to the *Configuring Evaporation Event* operating sequence on page 237 for more information.
3. This parameter appears only when the `Cleaning?` option has been configured in the **`PTV PHASE EVENTS`** menu. Refer to the *Configuring Cleaning Event* operating sequence on page 238 for more information.
4. An optional Back Flushing (BKF) system is available. If configured, it will be active during the injection and evaporation phases and also in the cleaning phase. Refer to *Enabling Backflush* operating sequence on page page 241 for details.
5. This line is displayed only when PTV Large Volume or PTV Solvent Split operating modes are used.

PTV Cryogenic Operation

An optional cryogenic system allows you to operate the PTV below ambient temperature using liquid nitrogen or liquid carbon dioxide as the coolant.

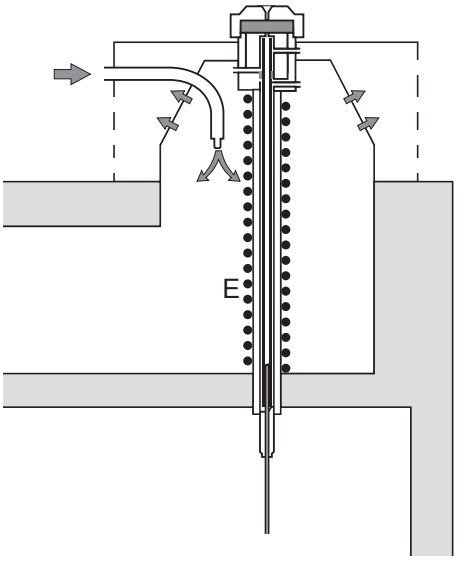
- liquid CO_2 allows PTV temperature down to -30 ºC.
- liquid N_2 allows PTV temperature down to -50 °C.

You can set the cryo system to operate during the **Prep Run** or **Post Run** phase.



E. Flow of Vapors of Liquid Nitrogen

Figure 11-9. Liquid Nitrogen Cooling System



E. Flow of Carbon Dioxide

Figure 11-10. Carbon Dioxide Cooling System

WARNING! High pressures and extremely low temperatures make liquid N_2 a hazardous material. High concentrations of N_2 in the air can cause an asphyxiation hazard. To avoid injury, always follow the safety precautions and delivery system design recommended by your gas supplier.

High pressures and extremely low temperatures make pressurized liquid CO_2 a hazardous material. High concentrations of CO_2 are dangerous. To avoid injury, always follow the safety precautions and delivery system design recommended by your gas supplier.

If your PTV has a cryogenic system, the **CONFIGURE INLET** menu contains the cryogenic configuration parameters. Each time you wish to use the cryogenic cooling option, you must enable it in the **CONFIGURE INLET** menu.

Without a cryogenic system, fans will cool the PTV to ambient temperature. With a PTV cryogenic system installed, you can specify a temperature at which the

cryogenic system switches on to cool the PTV. This temperature is the *cryo switch temp*.

Cryo Timeout

The *cryo timeout* feature allows you to limit the time the cryo system will run without receiving an injection signal. This serves two purposes:

- It conserves the cryogenic coolant.
- It turns off the cryo system if the setpoint temperature cannot be reached due to a lack of coolant.

After setting `Enable cryogenic?` to `Yes` in the **`CONFIGURE INLET`** menu, the cryo system begins automatically if you have turned on the `Auto prep run` feature the **`CONFIGURE OVEN`** menu. If `Auto prep run` is turned off, the cryo system begins when you press PREP RUN.

The cryo timeout will turn off the cryo system and reset the `Enable cryogenic?` parameter to `No` if the GC does not receive an injection signal by the time specified in the `Cryo timeout` parameter.

If this happens, you must re-enable the cryo system in the **`CONFIGURE INLET`** menu if you wish to perform an analysis using the PTV cryogenic system.

Refer to the *Configuring Cryogenic Operation* operating sequence on page 239 for instructions on using the cryo system.

PTV Backflush Operation

With the implementation of the backflush kit the TRACE GC Ultra equipped with the PTV injector, will be able to perform operations with the following advantages:

- Eliminate during the cleaning phase the heavy part of the sample, which are not relevant for the analysis. This will strongly reduce the analysis time with any analytical set-up and with many samples.

  This step is important when performing analysis of volatile compounds in a relatively low volatile mixture.
- Avoid solvent introduction into the column when performing a large volume injection. This is particularly important with MS applications.
- Perform precise cuts of the chromatogram, installing a selected coated precolumn, so that only a part of the sample is transferred into the column for the analysis.
- Use of very narrow bore column without significant peak broadening effect.
- In this way, for example, it is possible to use a thick film of stationary phase and to perform a precise cut of the components that are not of interest, so that is possible to analyze only the volatile compounds even with narrow bore capillary columns.

  The rest of the sample is eliminated through the injector and the oven temperature does not need to be increased to elevated value.

NOTE To install and configure the Backflush option refer to the Backflush System for PTV Injector Installation Guide.

The backflush principle of operation is schematically shown in Figure 11-11.

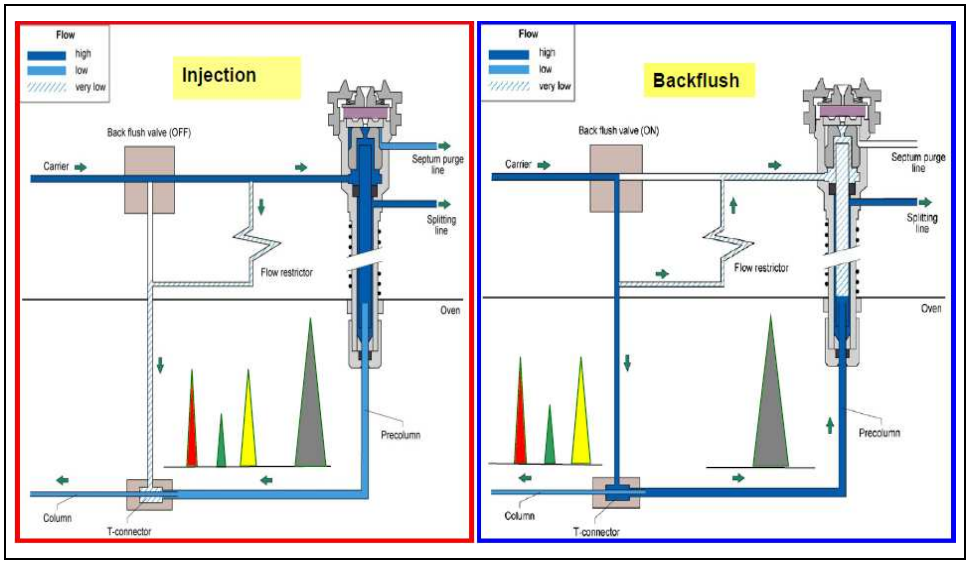


Figure 11-11. Backflush Kit for PTV Injector

Figure 11-11 shows the schematic flow diagram during the injection (left) and backflush (right) phase. While the analytes (red, green yellow peak) are transferred to the analytical column the slowly travelling high boilers (large grey peak) are still in the pre-column when the BKF is activated. Those matrix compounds are eliminated through the split line of the injector during the run time of the analysis. The analytes that have been transferred to the analytical column continue the regular chromatography.

Using Back Flushing

Backflushing can be applied only when the BEST PTV injector is used in the PTV operating modes such as PTV Split, PTV Splitless, PTV Solvent Split and PTV Large Volume

NOTE Backflush may be used during a large volume injection to avoid the solvent is entering into the column during venting phase.

When enabled, the Backflush is ON (solenoid valve not energized) during Injection Phase, Evaporation Phase and Cleaning Phase.

Large Volume Injections Using PTV

Large volume injection through a PTV can be done in different modes:

- **Mode 1***: At once in Solvent Split Mode (PTV LVI)*
  when the sample is introduced at a relatively high speed (e.g. over 10 µl/sec).
- **Mode 2***: Delayed Temperature Programming Splitless (DTPS)*
  when the sample is introduced at relatively high speed in splitless mode.
- **Mode 3***: Speed Controlled Injection in Solvent Split Mode (PTV LVI)*
  when the sample is introduced at a rate that is theoretically equal to the evaporation rate.
- **Mode 4***: Multiple injection*
  when a small volume of sample is introduced several times with a delay between the injections, each injection of about 5-10 µl.

With the BEST PTV, **mode 1**, **2** or **3** are used

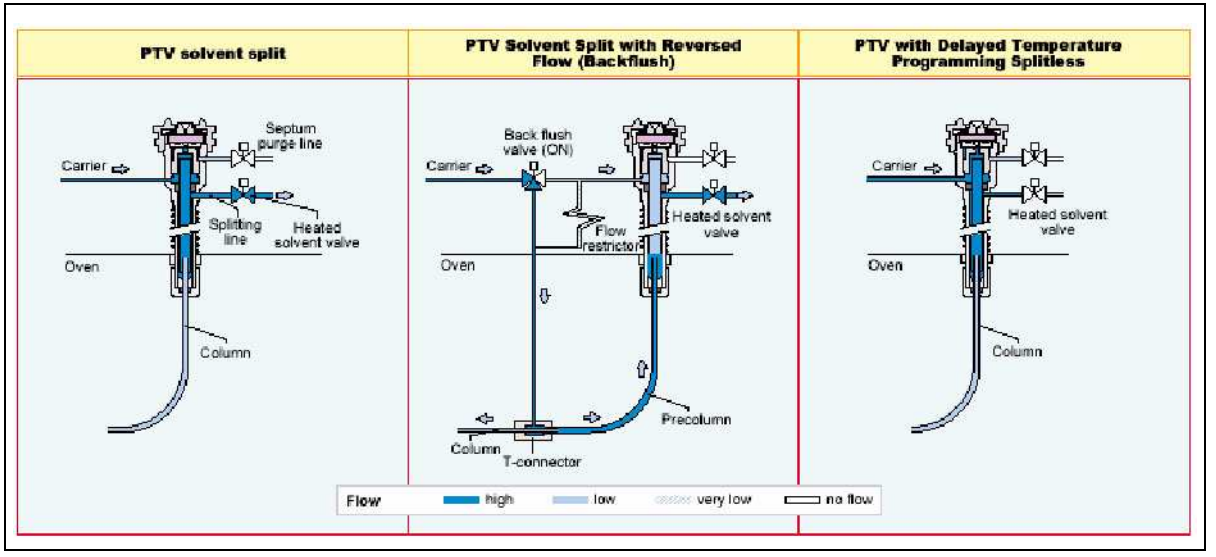
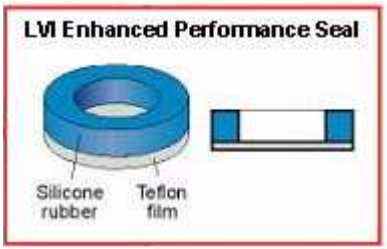


Figure 11-12. PTV Large Volume Injection Techniques



When performing Large Volume Injections with the TriPlus AS autosampler, it is strongly recommended to use LVI enhanced performance vial seals PN 313 010 01 (20 mm) or PN 313 011 02 (11 mm) or PN 313 010 03 (8 mm). These seal will avoid contamination with silicon, common to the conventional vial seals. These seals can be used for no more than 2-3 injections, depending on solvent volatility.

Mode 1: At once in Solvent Split Mode (PTV LVI)

It requires that the volume to be injected is not too large (normally below 80 µl) when using a 2 mm liner with silica wool.

- The **injection speed** must be relatively high (over 10 µl/sec) manually or with the autosampler.
- The **initial temperature** of the PTV must be normally kept (10-20 °C) below the boiling point of the solvent, corrected for the pressure in the injector. The temperature can be increased, to speed up the evaporation, but this can increase the loss of volatile compounds.

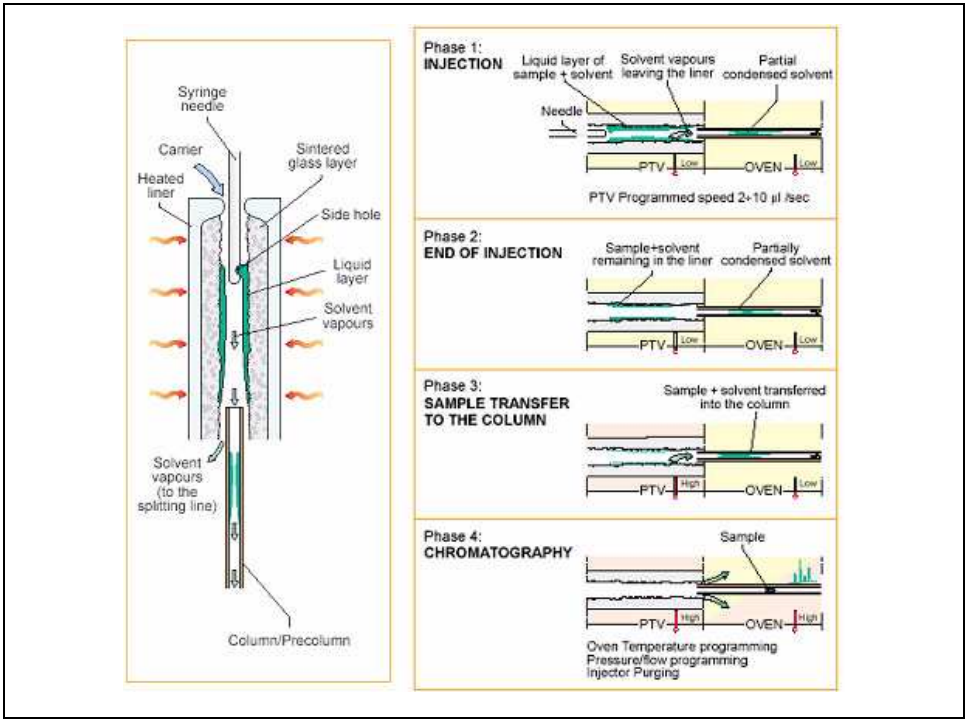
Solvent	B.P. Stand Cond	100 Kpa	200 Kpa	300 Kpa	400 Kpa	500 Kpa
Isopentane	28	49	65	77	87	96
Diethyl ether	35	54	72	84	93	101
n-Pentane	36	57	72	84	93	102
Dichloromethane	40	60	75	87	96	105
Methyl t-butyl ether	55	72	91	104	116	124
Methyl acetate	57	76	92	104	115	124
Chloroform	61	81	97	109	121	130
Methanol	65	82	97	107	116	124
n-Hexane	69	95	111	124	136	145
Ethyl acetate	77	97	114	126	136	145
Cyclohexane	81	106	122	137	149	159
Water	100	120	136	147	155	162

- The **split flow** must be in the range of 100-200 mL/min
- The **injection/evaporation time** depends on: the amount of solvent, the type of solvent and the amount of solvent that must remain in the liner before the injector is heated at the final temperature.

- The **final temperature** is selected according to the volatility of compounds to be transferred.
- The **oven temperature** must be selected according to the needed separation and to reduce the flooding into the column. Normally the boiling point of the solvent is a good starting point.

A certain amount of solvent in the liner must be left to increase the recovery for both the relatively volatile fraction (compounds at least with a boiling point 120 -150 °C higher then the solvent) and the rest of the sample. This amount should not flood significantly the column and must not create problems to the detector in use. To reduce the risk of column flooding, an empty deactivated precolumn (1-2 m) can be used in front of the coated column, unless Backflush option is used.

Mode 2: Delayed Temperature Programming Splitless (DTPS)



This technique is suited for the analysis of components (especially those with a boiling point close to the solvent boiling point) present in very low concentration.
In DTPS mode the injection is performed quite quickly (about 10 µl/sec) on a packed liner or on a glass sintered one (which requires a side hole needle syringe)
The PTV temperature is kept under the pressure corrected solvent boiling point during the injection phase.
The split valve and the purge valve are closed before the injection.
The PTV temperature can be increased just over the pressure corrected solvent boiling point during the evaporation phase, in order to transfer slowly the solvent (and the most volatile components) into the column, avoiding the generation of inlet overpressure (due to solvent evaporation) and avoiding the flooding of the column. During this phase the oven temperature is kept at the solvent boiling point at least until the end of the PTV transfer phase.
The duration of the evaporation phase depends on the amount of sample injected and the column flow: it can be 3-8 minutes for a 10-30 µl sample injection volume.
The PTV is then heated up to the transfer temperature in order to vaporize the components remained into the liner and transfer them into the column.
The duration of the transfer phase is generally 30-60 sec (as for a normal PTV splitless injection).
After the splitless transfer phase the split and the purge lines are open and the PTV temperature must be keep constant or increased to bake the injector.
The duration of the PTV evaporation phase, the temperature of the PTV during the evaporation, and the initial oven temperature must then be optimized considering the shape of the peaks in the chromatogram. If conditions are correctly chosen, normally is not required the installation of a precolumn.

Mode 3: Speed Controlled Injection in Solvent Split Mode (PTV LVI)

This is the injection mode that is normally used for the injection of large sample volumes. The TriPlus AS autosampler must be set in “D-Start Mode”. For instruction, refer to the *Operating Manual of the autosampler in use.*
The sample is injected at a ***controlled speed*** and this is normally in the range of 1-8 µl/sec according to the temperature/pressure and split flow in use.
The liquid sample is injected at a slow speed 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 but also on the type of packing present in the liner.
The injection mode permits the introduction of large amount of solvent (100-250 µl) because the solvent is not significantly stored in the liner and so the injectable volumes can be vary.

As for the ***At Once*** mode a certain amount of solvent must remain in the liner during the injection and after the injection before closing the split valve, to reduce the volatiles loss.
This technique anyway is not intended for the analysis of compounds with a boiling point close to that of the solvent.

PTV LVI Without the Back Flushing Device

Conditions that can be used to start the tuning are:

- Temperature close to the pressure corrected solvent boiling point (usually below that temperature).
- Split flow 100-150 mL/min.
- Injection speed 2-5 µL/sec.
- Injection/evaporation time 0.5-2 minutes after the injection end.
- Splitless time 0.25-1.5 min.
- Oven temperature slightly below the pressure corrected boiling point temperature of the solvent used.

The injection speed and the delay time after the injection are modified according to the chromatogram shape and peaks size, in comparison with a concentrated solution injected in PTV splitless mode.

The liner used is normally 2 mm ID with deactivated silica wool. If catalytic sensitive compounds have to be analyzed, a sintered glass liner must be used.
In this case the speed of injection and the maximum injection volume must be reduced (1-3 µls, 150 µl max) and a special syringe, with side hole needle, have to be installed in the autosampler.

PTVLVI With the Back Flushing Device

The kit for BKF LVI is required (PN 190 502 38).

With this mode of operation, the solvent elimination is strongly reduced and consequently the injection speed must be lower than 3 µl/sec. The amount of solvent remaining in the liner is usually higher and for this reason the precolumn must have a length of about 6 to 10 meters.

Conditions that can be used to start the tuning are:

- Temperature close to the pressure corrected solvent boiling point (usually below that temperature).
- Split flow 50-150 mL/min.
- Injection speed 1-3 µL/sec.
- Injection/evaporation time 0.1-1.5 minutes after the injection end.
- Splitless time 0.25-1.5 min.
- Oven temperature slightly below the pressure corrected boiling point temperature of the solvent used.

The injection speed and the delay time after the injection are modified according to the chromatogram shape and peaks size, in comparison with a concentrated solution injected in PTV splitless mode.

Glass sintered liner is recommended, but still the liner packed with deactivated silica wool can be also utilized. The use of the 51 mm needle with side hole syringe is required.

CAUTION When operating in Large Volume with backflush, program the closure of the septum purge during the whole transfer phase.
In the relevant PTV Control Table, scroll to `Const sept purge?` and press OFF/NO to deactivate constant septum purge flow. Scroll to `Stop purge for` and set the duration.

Temperature Profile and Timing

An example of temperature profile and timing in PTV large volume without and with backflush is shown respectively in Figures 11-13 and 11-14.

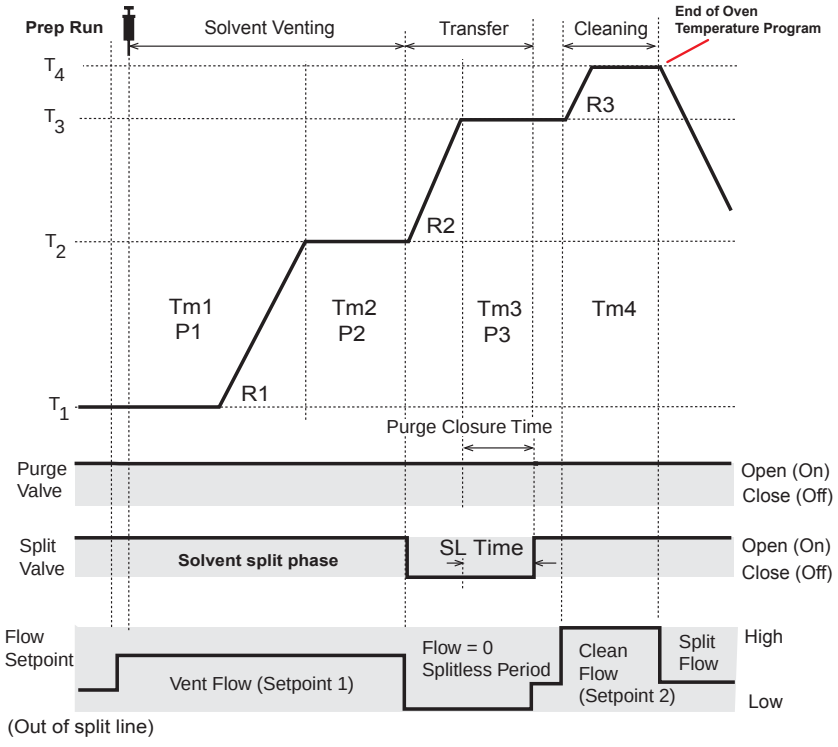
In Figure 11-13 (without Backflush) note that:

- the **purge valve** may be open or close according to the analytical requirements
- the **split valve** is closed at the end of evaporation phase and remain closed for the time programmed elapsed from the end of the *transfer ramp*.
- the **flow** through the split line is programmed to be changed during the *solvent vent phase* (see set point 1) and during the *cleaning phase* (see set point 2).

In Figure 11-14 (with Backflush) note that:

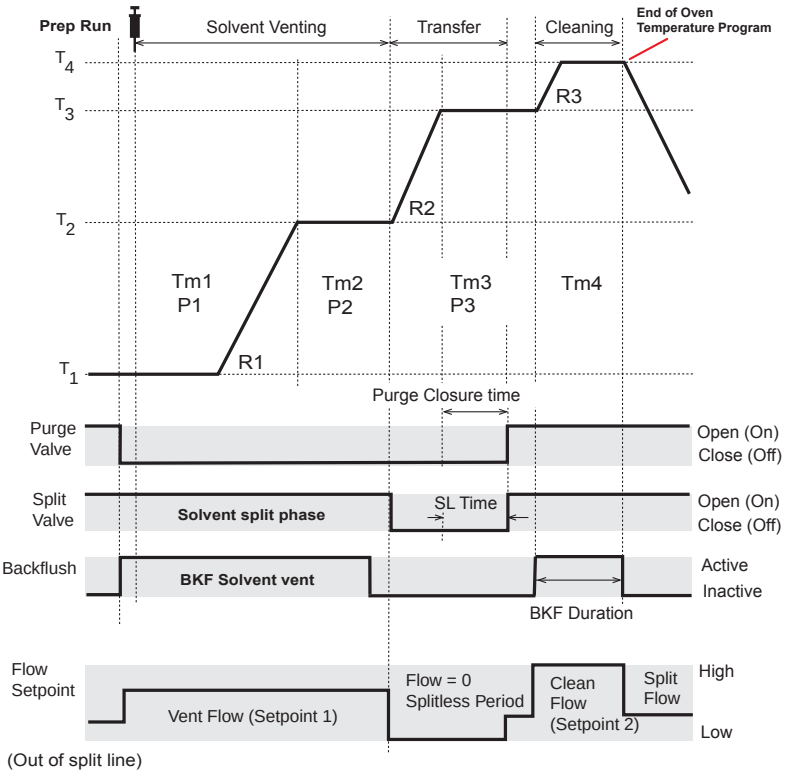
- The **purge valve** is closed at PREP RUN and remains closed for the time elapsed from the end of the *transfer ramp*.
- The **split valve** is closed at the end of evaporation phase and remain closed for the time programmed elapsed from the end of the *transfer ramp*.
- The **backflush valve** is active at PREP RUN and deactivated just before the transfer ramp, then activated at the beginning of the *clean ramp* and deactivated at the end of *clean time*.

The **flow** through the split line is programmed to be changed during the *solvent vent phase* (see set point 1) and during the *cleaning phase* (see set point 2).



T1. Injection Temperature	T3. Sample Transfer Temperature
T2. Evaporation Temperature	T4. Cleaning Temperature
Tm1. Injection Time	Tm2. Solvent Evaporation Time
Tm3. Transfer Time	Tm4. Cleaning Time
P1. Injection Pressure	P2. Evaporation Pressure
P3. Transfer Pressure	R1. Transfer Ramp
R2. Transfer Ramp	R3. Cleaning Ramp

Figure 11-13. Temperature Profile and Timing in PTV Large Volume Without Backflush



T1. Injection Temperature	T3. Sample Transfer Temperature
T2. Evaporation Temperature	T4. Cleaning Temperature
Tm1. Injection Time	Tm2. Solvent Evaporation Time
Tm3. Transfer Time	Tm4. Cleaning Time
P1. Injection Pressure	P2. Evaporation Pressure
P3. Transfer Pressure	R1. Transfer Ramp
R2. Transfer Ramp	R3. Cleaning Ramp

Figure 11-14. Temperature Profile and Timing in PTV Large Volume With Backflush Enabled

Further instructions on how to perform PTV LV Injections will be available in a separate instruction manual.

Example of Analysis with PTV Large Volume Injection

Figure 11-15, shown an example of PTV Large Volume injection (without backflush) of a hydrocarbons mixture (100 µL) performed in speed controlled mode. The analytical condition are reported in the table below the Figure 11-15.

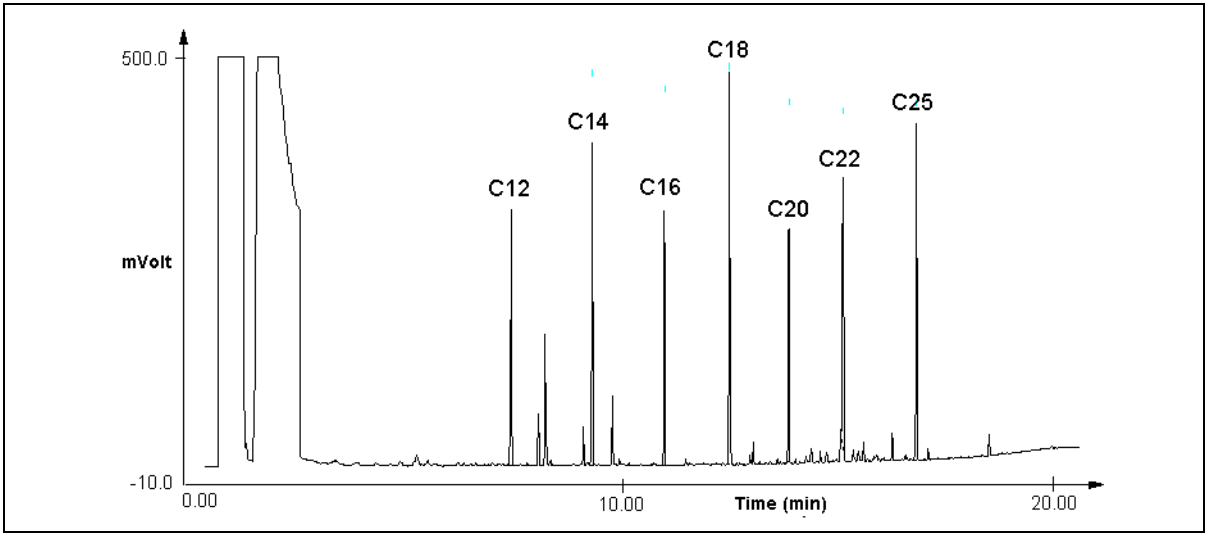


Figure 11-15. Example of PTV Solvent Split Large Volume Injection.

Analytical Conditions			
Sample	Hydrocarbons mixture (200 pg/µL) of C12 to C25 diluted in pentane		
Column	SE 52; 15 m length; 0.25 mm ID; 0.25 µm film thickness		
Carrier Gas	Helium; 1.2 mL/min	Oven Parameters	
Flow Mode	Constant Pressure	Initial Temperature 40 °C	Initial Time 2.00 min
Initial Press.	50 kPa	Final Temperature 310 °C	Hold Time 2.00 min
Liner	2 mm ID with Silica Wool	Rate 15.0 °C/min	
Injection Parameters	Injection Volume 100 µL	PTV Parameters	
	Injection Speed 5 µL/s	Base Temp. 30 °C	Splitless Time 1.00 min
Detector	FID	Solvent Valve Temp. 120 °C	Inject Time 0.3 min
Det. Base Temp.	320 °C	Vent Flow 100 mL/min	Transfer Rate 10 °C/sec
Det.Gas mL/min	H_2 35; Air 350; M-up 30	Transfer Temperature 275 °C	Transfer Time 15 min

Operating Sequences

OPERATING SEQUENCE

Installing a Liner and Septum

Materials required:

- liner
- septum
- spacer
- tweezers
- graphite seal
- screwdriver

WARNING! The injector fittings may be hot.
This sequence must be performed with the injector at room temperature.

1. Choose the correct liner for your application (see Table 11-1 on page 201). Slide a graphite seal, or the Viton® O-ring with adapter if a LVI with sintered glass liner is performed, onto the liner while gently turning the seal. Push it to 8–10 mm from the top of the liner. Viton® or Kalrez® O-ring can be used also for other glass liners listed in Table 11-1.

CAUTION Be careful not to break the graphite or allow graphite to enter in the liner.

2. Holding the top of the liner with tweezers, lower it into the injector. The liner should rest on the spacer at the bottom of the injector.

3. Insert the liner cap and secure it with the screwdriver. The liner cap must be screwed down tight enough to ensure a good seal between the liner and the injector body.

4. Place the septum support in the injector. The septum support must lie flush with the top of the injector. If not, the liner cap may not be tight enough.

5. Use tweezers to pick up the septum. Place the septum into the septum holder, then place the holder on top of the complete injector assembly.

CAUTION Use tweezers to pick up the septum to avoid contaminating it.

6. Gently finger-tighten the septum cap onto the injector assembly to hold the septum in place.

WARNING! Do not overtighten the septum cap. The septum will deform and may be difficult to penetrate with the syringe needle.

OPERATING SEQUENCE

Configuring Evaporation Event

Before you begin this sequence, configure the injector parameters.

1. Press CONFIG to enter the **CONFIGURE** menu.

2. Scroll to the inlet where your PTV injector is installed and press ENTER. The following menu appears:

CONFIG RIGHT INLET	
PTV phase events	<
Enable cryo	N

NOTE The `Enable cryo` parameter will be displayed only if your GC has a cryogenic system installed.

3. Scroll to `PTV phase events` and press ENTER to open the following menu:

PTV PHASE EVENTS	
Evaporation?	N<
Cleaning?	N

4. Scroll to `Evaporation?` and press YES. The relevant parameters will be displayed in the **INJECT PHASE** menu.

OPERATING SEQUENCE

Configuring Cleaning Event

1. Press CONFIG to enter the **CONFIGURE** menu.
2. Scroll to the inlet where your PTV injector is installed and press ENTER to open the following menu:

CONFIG RIGHT INLET	
PTV phase events	<
Enable cryo	N

NOTE The `Enable cryo` parameter will be displayed only if your GC has a cryogenic system installed.

3. Scroll to `PTV phase events` and press ENTER to open the following menu:

PTV PHASE EVENTS	
Evaporation?	N<
Cleaning?	N

4. Scroll to `Cleaning?` and press YES. The relevant parameters will be displayed in the **PTV PHASE** menu.

NOTE An optional back flush system prevents sample high boiled components from entering the analytical column during the cleaning phase. If installed, the back flushing valve will be active during the whole cleaning cycle.

OPERATING SEQUENCE

Configuring Cryogenic Operation

WARNING! High pressures and extremely low temperatures make liquid N_2 a hazardous material. High concentrations of N_2 in the air can cause an asphyxiation hazard. To avoid injury, always follow the safety precautions and delivery system design recommended by your gas supplier.

High pressures and extremely low temperatures make pressurized liquid CO_2 a hazardous material. High concentrations of CO_2 are dangerous. To avoid injury, always follow the safety precautions and delivery system design recommended by your gas supplier.

Use the following sequence to configure and enable the cryogenic system:

1. Press CONFIG to enter the **CONFIGURE** menu.
2. Scroll to the inlet where your PTV injector is installed and press ENTER to open the following menu:

```
      CONFIG RIGHT INLET
Enable cryogenic          Y<
Cryo Switch temp         50
Cryo timeout           0.10
Cool at             Prep run
```

3. Scroll to `Enable cryo` and press YES.
4. Scroll to `Cryo Switch temp` and enter the temperature at which the cryo system begins to operate.
5. Scroll to `Cryo timeout` and enter the time after a run starts that the cryo system should shut down if the GC does not receive an injection signal. Refer to *Cryo Timeout* on page 223.

6. Scroll to `Cool at` and press ON to open the following menu:

```
        INLET CRYO MODE
* Cool at prep run
  Cool at post run
```

Scroll to the mode you wish to select and press ENTER. An asterisk appears beside the selected cooling mode.

a. Select `Cool at prep run` to cool the injector at the beginning of the analytical cycle. The sample injection starts when the initial injector temperature is reached.

b. Select `Cool at post run` to cool the injector in the **Post Run** phase, during which the GC resumes the initial analytical conditions (including oven temperature and injector temperature). This option can save time between analyses.

OPERATING SEQUENCE

Enabling Backflush

Use the following sequence to configure and enable the backflush system:

1. Press CONFIG to enter the **CONFIGURE** menu.
2. Scroll to the inlet where your PTV injector is installed and press ENTER to open the following menu:

```
     CONFIG RIGHT INLET
PTV phase events
Enable cryo              N
Enable Back flush         Y<
```

3. Scroll with the ARROW key until the cursor points to `Enable back flush`. and set it `Y`.
4. Press CLEAR to return the main **CONFIGURE** menu.

IMPORTANT! Backflush may be manually activated On/Off through the **VALVES** menu (press VALVES). It is very useful when the liner replacement is required, particularly when a MS is used.

During column evaluation the Backflush is ON.
To perform column evaluation when Backflush is not used, it must be disabled and the line in the GC oven must be sealed with a metal pin and relevant seal (PN 290 034 97).

OPERATING SEQUENCE

Programming the PTV Split Mode

In PTV split mode, the split and purge valves remain open during an entire run.

Before you begin this sequence, do the following:

- Verify that a column is correctly installed, the correct liner is in the injector, and the system is free of leaks.
- Check the oven temperature and detector temperature.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.

1. Press RIGHT INLET to open the **`RIGHT INLET (PTV)`** menu.
2. Scroll to `Mode:` and press MODE/TYPE. In the **`INLET MODE`** submenu, scroll to `PTV Split` and press ENTER.
3. Scroll to `Temp` and set the appropriate injector temperature.
4. Specify the split flow or split ratio. To set the split flow, scroll to `Split flow` and enter the value in mL/min. The split ratio will be calculated for you.

   To set the split ratio, scroll to `Split ratio` and enter that value. The split flow will be calculated for you.

NOTE

The split ratio is the ratio between the split flow and the column flow. For example, if the column flow is 2 mL/min, a 50 mL/min split flow gives a split ratio of 25:1. Only 1/25 of the injected sample would enter the column. The `Split ratio` calculates the split flow from the column flow used during the Prep Run phase.

5. Scroll to `Inject phase menu` and press ENTER to open the **`INJECT PHASE MENU`** or press RAMP # to jump to the various programmed phases.
   If you want to program temperature ramps, refer to the *Programming Injection Parameters* operating sequence on page 247 for instructions.

OPERATING SEQUENCE

Programming the PTV Splitless Mode

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 (SL) programmed.

Before you begin, do the following:

- Verify that a column is correctly installed, the correct liner is in the injector, and the system is free of leaks.
- Check the oven temperature and detector temperature.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.

1. Press RIGHT INLET to open the **`RIGHT INLET (PTV)`** menu.
2. Scroll to `Mode:` and press MODE/TYPE. In the **`INLET MODE`** submenu, scroll to `PTV Splitless` and press ENTER.
3. Scroll to `Temp` and set the appropriate injector temperature.
4. Scroll to `Split flow` and enter the desired value in mL/min.
5. Scroll to `Splitless time` and enter the time during which the split valve should be closed.
6. If constant septum purge is required, scroll to `Const sept purge?` and press YES. If constant septum purge is not required, keep `Const sept purge?` set to `No`, then scroll to `Stop purge for` and enter the duration.

If you want to program temperature ramps, refer to the *Programming Injection Parameters* operating sequence on page 247 for instructions.

OPERATING SEQUENCE

Programming the PTV in DTPS Mode

In PTV Delayed Temperature Programming Splitless (DTPS) mode, the split and purge valves are closed during the **Prep Run** phase and remain closed up to the end of the transfer time (SL) programmed, usually from 3 to 8 minutes. Consider that with DTPS the initial time must be adequate to allows solvent entering into the column before heating the PTV to the transfer temperature.

Before you begin, do the following:

- Verify that a column is correctly installed, the correct liner is in the injector, and the system is free of leaks.
- Check the oven temperature and detector temperature.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.

1. Press RIGHT INLET to open the **`RIGHT INLET (PTV)`** menu.
2. Scroll to `Mode:` and press MODE/TYPE. In the **`INLET MODE`** submenu, scroll to `PTV Splitless` and press ENTER.
3. Scroll to `Temp` and set the appropriate injector temperature.
4. Scroll to `Split flow` and enter the desired value in mL/min.
5. Scroll to `Splitless time` and enter the time during which the split valve should be closed.
6. Scroll to `Const sept purge?` and press NO then scroll to `Stop purge for` and enter the duration.

If you want to program temperature ramps, refer to the *Programming Injection Parameters* operating sequence on page 247 for instructions.

OPERATING SEQUENCE

Programming the PTV Solvent Split Mode

In PTV solvent split 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 (SL) programmed.

Before you begin, do the following:

- Verify that a column is correctly installed, the correct liner is in the injector, and the system is free of leaks.
- Check the oven temperature and detector temperature.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.

1. Press RIGHT INLET to open the **`RIGHT INLET (PTV)`** menu.
2. Scroll to `Mode:` and press MODE/TYPE. In the **`INLET MODE`** submenu, scroll to `PTV Solvent split` and press ENTER.
3. Scroll to `Temp` and set the appropriate injector temperature.
4. Scroll to `Split flow` and enter the desired value in mL/min.
5. Scroll to `Splitless time` and enter the time during which the split valve should be closed.
6. Scroll to `Const sept purge?` and press ON/YES to activate a constant septum purge flow, if required. If constant septum purge is not required, keep `Const sept purge?` set to `No`, then scroll to `Stop purge for` and set the duration.
   If you want to program temperature ramps, refer to the *Programming Injection Parameters* operating sequence on page 247 for instructions.

OPERATING SEQUENCE

Programming the PTV Large Volume Mode

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 at the end of the injection time and evaporation time, if programmed. It remains closed up to the end of the transfer time (SL) programmed.

Before you begin, do the following:

- Verify that a column is correctly installed, the correct liner is in the injector, and the system is free of leaks.
- Check the oven temperature and detector temperature.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.

1. Press RIGHT INLET to open the **`RIGHT INLET (PTV)`** menu.
2. Scroll to `Mode:` and press MODE/TYPE. In the **`INLET MODE`** submenu, scroll to `PTV large volume` and press ENTER.
3. Scroll to `Temp` and set the appropriate injector temperature.
4. If you want a specific split flow, scroll to `Split flow` and enter that value.
5. Scroll to `Splitless time` and enter the time during which the split valve should be closed.
6. Scroll to `Solvent vlv` and set the appropriate solvent valve temperature.
7. Scroll to `Const sept purge?` and press ON/YES activate a constant septum purge flow, if required. If constant septum purge is not required, keep `Const sept purge?` set to `No`, then scroll to `Stop purge for` and enter the duration.

If you want to program temperature ramps, refer to the *Programming Injection Parameters* operating sequence on page 247 for instructions.

NOTE If a PTVLVI with Backflush system is required, the backflush valve must be enabled as described on page 241 and a BKFLVI optional kit is suggested.

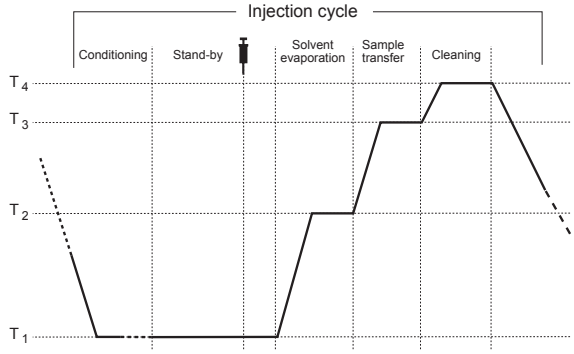
OPERATING SEQUENCE

Programming Injection Parameters

Use the following sequence to program temperature ramps when operating in PTV split, PTV splitless, PTV DTPS, PTV solvent split, or PTV large volume mode. Be sure to program the other operating mode parameters before programming the temperature ramps.

PTV Injection Cycle

A generic temperature program of the PTV injection cycle is shown in Figure 11-16.



T1. Injection Temperature
T2. Evaporation Temperature
T3. Sample Transfer Temperature
T4. Cleaning Temperature

Figure 11-16. Generic Temperature Profile

1. In the **RIGHT INLET (PTV)** menu, scroll to `Inject phase menu` and press ENTER to open the **INJECT PHASE MENU**.

2. Scroll to `Ramped pressure?`. Press YES to program ramped pressure.
3. Scroll to `Inject pres` and enter the injection phase pressure at the beginning of the temperature ramp.
4. Scroll to `Inject temp` and enter an injector temperature lower than the solvent boiling point at the programmed pressure.
5. Scroll to `Inject time` and set the time the injector temperature must be maintained.
6. Scroll to `Transfer pres` and set the sample transfer phase pressure.
7. Scroll to `Transfer temp` and set the sample transfer temperature.
8. Scroll to `Transfer ramp` and set the rate in ºC/s to reach the sample transfer temperature.
9. Scroll to `Transfer time` and set the time the transfer temperature must be maintained.

WARNING! Consider a time almost similar to the oven temperature program unless the cleaning phase is used.

10. Scroll to `Vent flow` (only in PTV Large Volume and PTV Solvent Split modes) and set the vent flow required during the injection and evaporation phases.

If Solvent Evaporation Has Been Configured:

1. Scroll to `Evap pres` and set the initial pressure for the evaporation temperature ramp during the solvent evaporation phase.
2. Scroll to `Evap ramp` and set the rate in ºC/s to reach the solvent evaporation temperature.
3. Scroll to `Evap temp` and set the solvent evaporation temperature.
4. Scroll to `Evap time` and set the time the transfer temperature must be maintained.

If Injector Cleaning Has Been Configured:

1. Scroll to `Cleaning temp` and set the injector cleaning temperature.
2. Scroll to `Cleaning ramp` and set the rate in ºC/s to reach the cleaning temperature.
3. Scroll to `Cleaning time` and set the time the cleaning temperature must be maintained.
4. Scroll to `Clean flow` and set the value to increase the flow during the cleaning phase.

The `Inject pres`, `Transfer pres`, and `Evap pres` parameters will not be displayed if `Ramped pres` is set to `No`.
If `Ramped pres` is set to `Yes` in Inject Phase Menu, please read the considerations reported in *Ramped Pressure Option in Menu Inject Phase Menu*.

If the Back Flushing system (BKF) is available, the BKF valve is activated at the beginning of the cleaning phase and remains active up to the end of the cleaning time.

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 in Carrier Gas Menu, 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, the control of the carrier will return to be managed through the Carrier Gas Menu. This means that the pressure/flow will be back to the value defined in the Carrier Gas Menu for that moment of the analysis. If *programmed pressure/flow* 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 and the **BKF is not enabled**, 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).
If the **BKF is enabled** during the cleaning, the flow will be reversed, therefore this phase cannot be used for maintaining the injector temperature. Transfer time duration must then be long as the whole oven temperature program and consequently the pressure will maintain the value defined in Transfer Pressure and the pressure/flow program defined in the Carrier Gas Menu will not be executed.

OPERATING SEQUENCE

Programming the CT Split Mode

In CT split mode, the split and purge valves remain open throughout the run.

Before you begin, do the following:

- Verify that a column is correctly installed, the correct liner is in the injector, and the system is free of leaks.
- Check the oven temperature and detector temperature.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.

1. Press RIGHT INLET to open the **`RIGHT INLET (PTV)`** menu.
2. Scroll to `Mode:` and press MODE/TYPE. In the **`INLET MODE`** submenu, scroll to `CT Split` and press ENTER.
3. Scroll to `Temp` and set the appropriate injector temperature.
4. Specify the split flow or split ratio. To set the split flow, scroll to `Split flow` and enter the value in mL/min. The split ratio will be calculated for you. To set the split ratio, scroll to `Split ratio` and enter that value. The split flow will be calculated for you.

NOTE The split ratio is the ratio between the split flow and the column flow. For example, if the column flow is 2 mL/min, a 50 mL/min split flow gives a split ratio of 25:1. Only 1/25 of the injected sample would enter the column. The `Split ratio` calculates the split flow from the column flow used during the Prep Run phase.

OPERATING SEQUENCE

Programming the CT Splitless Mode

In CT splitless mode, the split and purge valves are closed during the **Prep Run** phase and remain closed after the injection for the programmed duration.

Before you begin, do the following:

- Verify that a column is correctly installed, the correct liner is in the injector, and the system is free of leaks.
- Check the oven temperature and detector temperature.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.

1. Press RIGHT INLET to open the **`RIGHT INLET (PTV)`** menu.
2. Scroll to `Mode` and press MODE/TYPE. In the **`INLET MODE`** submenu, scroll to `CT Splitless` and press ENTER.
3. Scroll to `Temp` and set the appropriate injector temperature.
4. Scroll to `Split flow` and enter the desired value in mL/min.
5. Scroll to `Splitless time` and enter the time during which the split valve should be closed.
6. Scroll to `Const sept purge?` and press ON/YES to activate a constant septum purge flow, if required.

   If constant septum purge is not required, keep `Const sept purge?` set to `No`, then scroll to `Stop purge for` and enter the duration.

OPERATING SEQUENCE

Programming the CT Surge Splitless Mode

In CT surge splitless mode, a carrier gas pressure surge activates during the injection phase for a programmed time. This 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 you begin, do the following:

- Verify that a column is correctly installed, the correct liner is in the injector, and the system is free of leaks.
- Check the oven temperature and detector temperature.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.

1. Press RIGHT INLET to open the **`RIGHT INLET (PTV)`** menu.
2. Scroll to `Mode` and press MODE/TYPE. In the **`INLET MODE`** submenu, scroll to `CT Splitless w/srg` and press ENTER.
3. Scroll to `Temp` and set the appropriate injector temperature.
4. If you want a specific split flow, scroll to `Split flow` and enter that value.
5. Scroll to `Splitless time` and enter the time during which the split valve should be kept closed.
6. Scroll to `Surge pressure` and enter the pressure surge value.Scroll to `Surge duration` and enter the pressure surge duration.
7. Scroll to `Const sept purge?` and press ON/YES to activate a constant septum purge flow, if required. If constant septum purge is not required, keep `Const sept purge?` set to `No`, then scroll to `Stop purge for` and enter the duration.

12

Gas Sampling Valve (GSV)

This chapter describes the gas sample valve available with the TRACE GC Ultra and contains operating sequences for automatic sampling.

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GSV Overview 253
Automatic GSV Menus 254

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Programming an Automatic Single Sampling 256
Programming an Automatic Multi Sampling 257
Performing a Single Injection with the Automatic Sampling Valve 258
Performing a Multi Injection with the Automatic Sampling Valve 259

GSV Overview

For automatic gas sampling, a 6-port Valco valve is used. A wide range of sampling loops allows the injections of different volume of samples.

The valve is installed on the top of the GC. The valve is not heated.

The sampling loop is installed on the valve. The sample inlet and outlet connecting ports are located on the rear of the GC.
A filter is installed on the **load sample** line to prevent that suspension particles may damage the valve.

The switching between **load sample** and **inject sample** positions (and vice-versa) is controlled through the TRACE GC Ultra keypad.

Automatic GSV Menus

Gas sampling valve editor has two menus according to single or multi sampling options.

Menu for Single Sampling

Press VALVES to open the **VALVES** menu.

```
        VALVES

Inlet Valves
# 2 Gas sample   Load<
```

Scroll to #2 `Gas sample` then press ENTER to open the **SAMPLING VALVE** submenu.

```
    SAMPLING VALVE

* Load     <
  Inject
```

Scroll to the valve position you want to set as default. Press ENTER to confirm the selection. An asterisk appears on the left of the valve position selected.

Table 12-1. Single Sampling Menu

Menu	Submenu	Comments for Menu
VALVES		This line is the menu title bar.
#2 Gas sample	SAMPLING VALVE Load Inject	The range is Inj=On, Load=Off

Menu for Multi Sampling

Press RUN TABLE to open the **RUN TIME EVENTS** menu.

```
    RUN TIME EVENTS
0.00 Valve #2     Inj <
Add run time event
Ext. event defaults
```

Scroll to `0.00 Valve #2` then press ENTER to open the **RUN TIME EVENTS** submenu.

```
    RUN TIME EVENTS
Valve # Sampling
Inject at          0.00<
Inject for         1.00
```

Set the time at which the injection must begin and set the time the sampling valve must be maintained on the inject position.

Table 12-2. Multi Sampling Menu

Menu	Submenu	Comments for Submenu
RUN TIME EVENTS		This line is the menu title bar.
0.00 Valve #2	RUN TIME EVENTS	
	Valve # Sampling	This line is the submenu title.
	Inject at	This indicates the time at which the injection must begin.
	Inject for	This indicates the time the sampling valve must be maintained on inject position.

OPERATING SEQUENCE

Programming an Automatic Single Sampling

Before programming the single gas sampling option, do as follows:

- Verify that a column is correctly installed, and the system is free of leaks.
- Check the oven temperature and detector temperature.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.

WARNING! Hydrogen is a potentially dangerous gas. Refer to *Using Hydrogen* on page xxviii for safety information.

1. Press VALVES to open the **VALVES** menu.
2. Scroll to #2 `Gas sample`
3. Press ENTER to open the **SAMPLING VALVE** submenu.
4. Scroll to the valve position `Load` or `Inject` you want to set as default.
5. Press ENTER to confirm the selection.

OPERATING SEQUENCE

Programming an Automatic Multi Sampling

Before programming the multi gas sampling option, do as follows:

- Verify that a column is correctly installed, and the system is free of leaks.
- Check the oven temperature and detector temperature.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.

WARNING! Hydrogen is a potentially dangerous gas. Refer to *Using Hydrogen* on page xxviii for safety information.

1. Press RUN TABLE to open the **RUN TIME EVENTS** menu.
2. Scroll to `0.00 Valve #2.`
3. Press ENTER to open the **RUN TIME EVENTS** submenu.
4. Scroll to `Inject at` and enter the time at which the injection must begin.
5. Scroll to `Inject for` and enter the time the sampling valve must be maintained on inject position.

OPERATING SEQUENCE

Performing a Single Injection with the Automatic Sampling Valve

Before injecting the sample, do the following:

- Verify that the column and liner, if used, or adapter are correctly installed and the system is free of leaks.
- Check the oven temperature and detector temperature.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.

WARNING! Hydrogen is a potentially dangerous gas. Refer to *Using Hydrogen* on page xxviii for safety information.

1. Press VALVES to open the **VALVES** menu.
2. Scroll to #2 `Gas sample`
3. Press ENTER to open the **SAMPLING VALVE** submenu.
4. Scroll to the valve position `Load` then press ENTER to confirm the selection.
5. Press PREP RUN.
6. When the **Ready to Inject** LED is lit, fill the sampling loop.
7. When the sampling loop is filled, scroll to the valve position `Inject` then press ENTER to confirm the selection.
8. Press START.
9. After the time necessary for the sample transfer, scroll to the valve position `load` then press ENTER to confirm the selection.

The GC will complete the analysis as programmed.

OPERATING SEQUENCE

Performing a Multi Injection with the Automatic Sampling Valve

Before injecting the sample, do the following:

- Verify that the column and liner, if used, or adapter are correctly installed and the system is free of leaks.
- Check the oven temperature and detector temperature.
- Program the carrier gas flow as described in Chapter 4, *Digital Gas Control*.

WARNING! Hydrogen is a potentially dangerous gas. Refer to *Using Hydrogen* on page xxviii for safety information.

1. Press RUN TABLE to open the **RUN TIME EVENTS** menu.
2. Scroll to `0.00 Valve #2`
3. Press ENTER to open the **RUN TIME EVENTS** submenu.
4. Scroll to `Inject at` and enter the time at which the injection must begin.
5. Scroll to `Inject for` and enter the time the sampling valve must be maintained on inject position.
6. Press PREP RUN.
7. When the **Ready to Inject** LED is lit, fill the sampling loop. When the sampling loop is filled, press CONFIG and select `Oven` to open **OVEN** menu.
8. In **OVEN** menu, set both `Auto prep` run and `Autostart` ON.

The GC will complete the analysis as programmed, then the sampling cycle is automatically repeated. Pay attention to fill the sampling loop with a new sample at the begin of each sampling cycle otherwise the same sample will be continuously loaded.

SECTION

IV

The Oven and Columns

This section contains information about the configuration options for the TRACE GC Ultra column oven and procedures for using capillary and packed columns in the oven.

Chapter 13, *The Column Oven*, describes the features and configuration options for the TRACE GC Ultra column oven and includes operating sequences for oven programming.

Chapter 14, *Columns*, describes the analytical columns used in the TRACE GC Ultra. The operating sequences for leak test and column evaluation are also included.

13

The Column Oven

This chapter describes the features and configuration options for the TRACE GC Ultra column oven and includes operating sequences for oven programming.

WARNING! When UFM Device is installed on your GC, the relevant column oven features and configuration options are described in the UFM Ultra Fast Module Instruction Manual.

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Oven Menu 272

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Setting Up a Single Ramp Temperature Program 274
Setting Up a Multiple Ramp Temperature Program 275

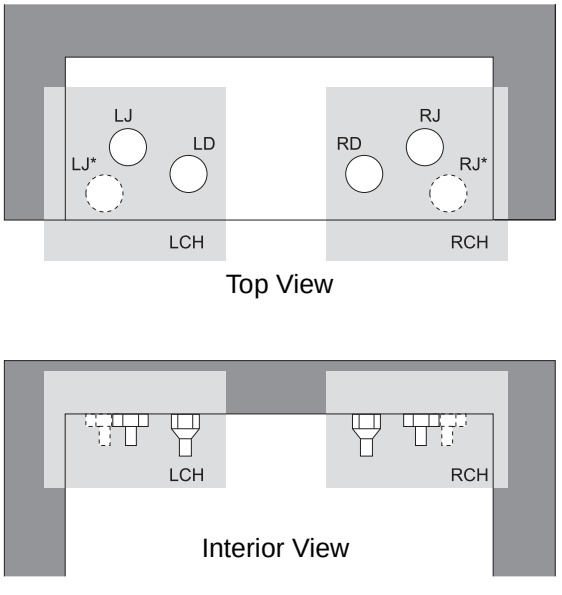
Column Oven Overview

The TRACE GC Ultra column oven provides a stable heating environment for the analytical column. The oven heats and cools quickly. Efficient air circulation ensures a high degree of thermal stability.

Opening the oven door activates a safety microswitch, which automatically switches off the oven heating and the motor for the air circulation fans. The oven is heated by resistor elements powered by a circuit located within the GC control unit.

The column fittings in the oven depend on whether capillary or packed column injectors and detector base bodies are installed. Auxiliary gas lines, if installed, end in M8x1 male fittings between the injector and the detector base bodies. The oven temperature is monitored by a PT 100 platinum wire sensor and controlled by the GC control unit.

Figure 13-1 shows the left and right detector and injector positions on top of the oven and the fittings inside the oven.



LCH (Left Channel)	RCH (Right Channel)
LJ Left Injector	RJ Right Injector
LJ* 2nd Left Injector	RJ* 2nd Right Injector
LD Left Detector	RD Right Detector

Figure 13-1. Injector/Detector Locations and Fittings

The column oven has the following capabilities:

- maximum temperature of 450 °C
- maximum temperature increase rate of 120 °C/min
- seven linear temperature ramps
- minimum operating temperature of a few degrees above ambient, which is obtained by two modulated cooling flaps controlled by the GC, shown in Figure 13-2

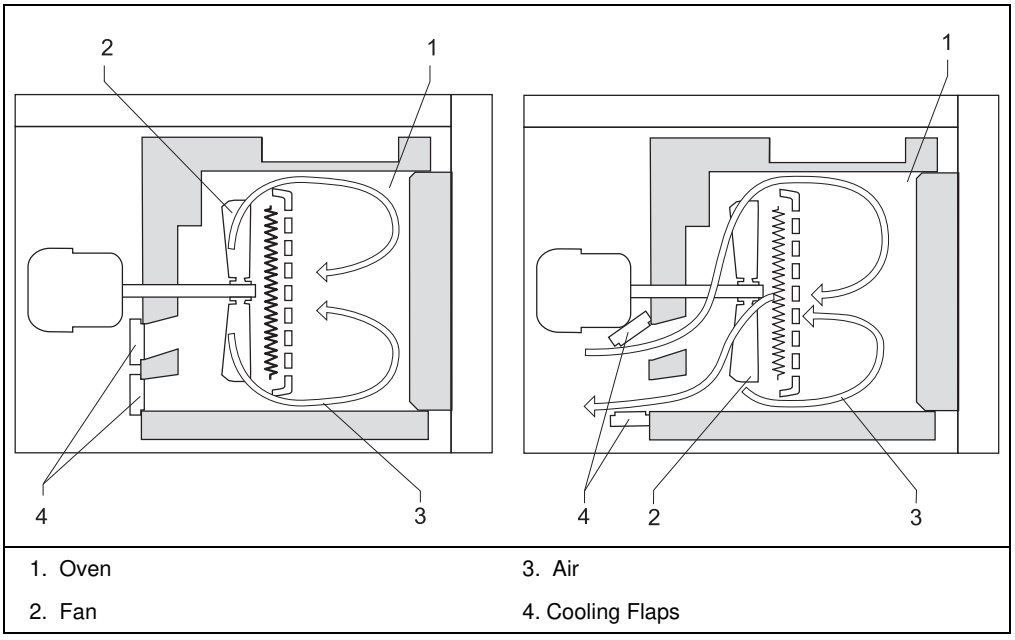
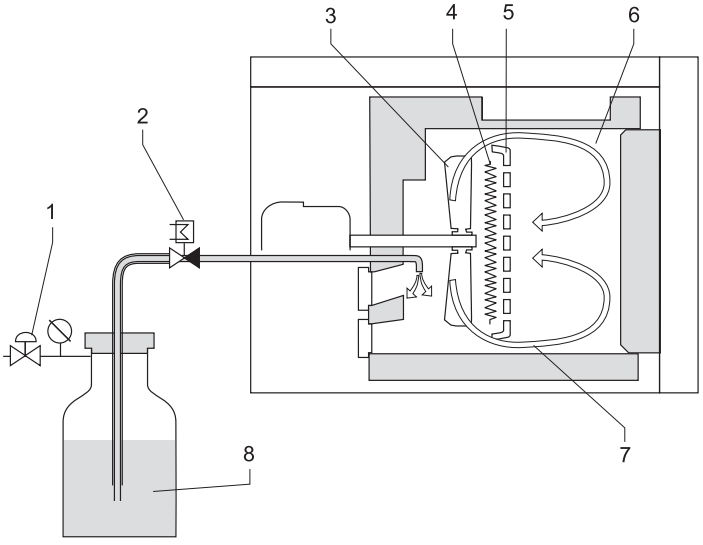


Figure 13-2. Oven Air Circulation

- temperature control through:
  - — heater control
  - — fine control of hot air exhaust
  - — ambient air intake

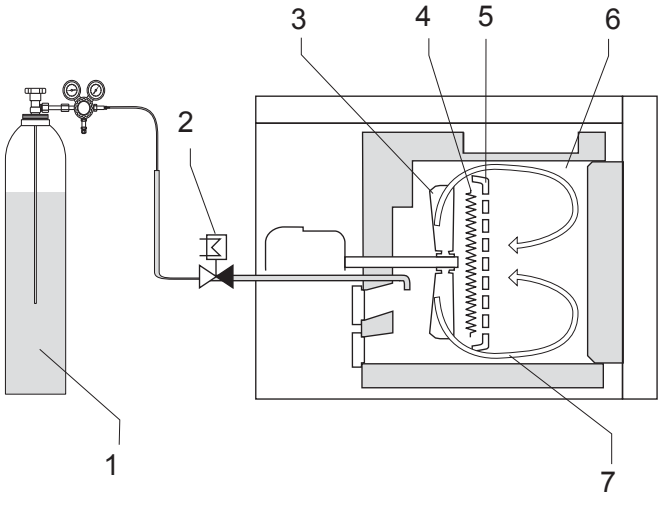
- separation of moderately volatile components on thick film capillary columns at near ambient temperatures without the use of a cryogenic system
- with a cryogenic option installed, the oven temperatures can reach –55 °C with liquid carbon dioxide or –99 °C with liquid nitrogen

Figures 13-3 and 13-4 show the cryogenic system with liquid nitrogen and liquid dioxide as a coolant. When liquid carbon dioxide is used, the cylinder must have a dip tube. Refer to the *Site Preparation and Installation Manual* for more information about connecting cryogenic coolants.



1. Pressure Regulator for Liquid N_2	5. Shield
2. Dosing Valve	6. Oven
3. Fan	7. Air
4. Heater Back	8. Coolant Reservoir for Liquid N_2

Figure 13-3. Cryogenic System with Liquid Nitrogen



1. Coolant Reservoir for Liquid CO_2
2. Dosing Valve
3. Fan
4. Heater Back
5. Shield
6. Oven
7. Air

Figure 13-4. Cryogenic System with Liquid Carbon Dioxide

Oven Safety

Opening the oven door cuts off the power to the oven heater, fan, and the cryogenic system (if installed). The setpoints are kept in memory. The display shows the following safety message:

```
            OVEN

Temp          40  Door Open

Initial time           2.00

Ramp 1                  Off
```

To return to normal operation, close the oven door.

WARNING! The oven vents at the rear of the GC discharge hot air during cooling.

Hydrogen is a potentially dangerous gas. When hydrogen is used as a carrier gas, the column oven must have a hydrogen sensor. Refer to *Using Hydrogen* on page xxviii for hydrogen safety information.

For safety information about liquid carbon dioxide and liquid nitrogen, refer to *Using Liquid Coolants* on page xxx.

Column Oven Configuration

The **CONFIGURE OVEN** menu contains the control parameters for the column oven.

Press CONFIG, then scroll to `Oven` and press ENTER to open the menu shown in Table 13-1. Refer to Chapter 3, *Configuration*, for more information about the **CONFIGURE** menu.

Table 13-1. Configure Oven Menu

Menu	Range	Comments
`CONFIGURE OVEN`		This line is the menu title bar.
`Auto prep run`	On/Off	Press ON to turn on automatic **Prep Run** execution without pressing PREP RUN. This feature is useful when you use the autosampler. When this item is set to `Off`, you must press PREP RUN to activate the **Prep Run**.
`Auto Start`	On/Off	Allows an automatic *Start* signal.
`PR timeout`	0–999.99 min, ∞	Enter the duration of the prep run. The injection must occur within this time or the timeout will return the GC to the **Standby** condition.
`Equil time`	0–999.99 min	This is the time required for equilibrating the oven temperature after the temperature is set or modified.
`Ready delay`	0–99.9 min	This parameter delays the **Ready to Inject** condition. This function is useful for on-column injectors. It allows the secondary cooling to cool the injector before the injection. This time must not exceed the `PR timeout` value.
`Max temp`	0–450 °C	This parameter defines the maximum allowable oven temperature setpoint to protect the column from unintentionally high temperatures. This limit must be set to the manufacturer's maximum recommended operating temperature for the column.

Table 13-1. Configure Oven Menu (Continued)

Menu	Range	Comments
`Enable cryogenics`[1]	Yes/No	This function enables the oven's cryogenic system when it is installed and configured with CO_2 or LN_2 as a coolant. Press YES to activate the cryogenic system. Press NO to deactivate it.
`Cryo Timeout`[1]	0–999.99 min	This parameter specify the time at which the cryo system will be disabled. This parameter is active during the "cooling "and the "stand-by" phases.
`Start cryo at`[1]	40 to 200 °C	This parameter specifies the temperature at which the cryo system begins to supply the coolant.

1. If the cryogenic system is installed and configured, its parameters are included in the menu. If the UFM option is installed, these parameters are not available for user modification.

OPERATING SEQUENCE

Configuring the Column Oven

Use this sequence to configure the column oven.

Configuration Without the Cryogenic System

1. Press CONFIG, then scroll to `Oven` and press ENTER.
2. Scroll to `Auto prep run`. Press ON to enable automatic prep run. Press OFF if you want the prep run to be activated by pressing the PREP RUN key.
3. Scroll to `PR timeout` and set the duration of the prep run timeout.
4. Scroll to `Equil time` and set the oven temperature equilibration time.
5. Scroll to `Ready delay` and set the delay time before the GC enters the **Ready to Inject** condition.
6. Scroll to `Max Temp` and set the maximum allowable oven temperature.

Configuration with the Cryogenic System

1. Press CONFIG, then scroll to `Oven` and press ENTER.
2. Scroll to `Auto prep run`. Press ON to enable automatic prep run. Press OFF if you want the prep run to be activated by pressing the PREP RUN key.
3. Scroll to `PR timeout` and set the duration of the prep run timeout.
4. Scroll to `Enable cryogenics` and press YES to enable the cryogenic system or NO to disable it.

If the UFM option is installed with the cryo oven, the `Enable cryogenics` menu option is not available to the user because the cryo variables are automated.

5. Scroll to `Cryo timeout` and enter the time at which the cryo system will be disabled. This parameter is active during the “cooling” and the “stand-by” phases.
6. Scroll to `Cool at` and specify the temperature. at which the cryo system begins to supply the coolant.
7. Scroll to `Equil time` and set the oven temperature equilibration time.
8. Scroll to `Ready delay` and set the delay time before the GC enters the **Ready to Inject** condition.
9. Scroll to `Max Temp` and set the maximum allowable oven temperature.

Oven Menu

The **OVEN** menu contains the parameters for programming the oven temperature, from an initial temperature to a final temperature, using up to seven ramps during the analytical run. It is possible to set a single (isothermal) or multiple ramp program.

Press OVEN to open the **OVEN** menu, shown in Table 13-2.

Table 13-2. Oven Menu

Menu	Range	Comments
OVEN		This line is the menu title bar.
Temp	On/Off, 0–450 °C[1]	Press ON to display the actual and setpoint values. This value is the initial temperature.
Initial time	0–999.99 min	This parameter defines the time the oven remains at the starting temperature after a programmed run has begun.
Ramp 1	On/Off, ∞ 0.0–120 °C/min	This is the temperature ramp rate in °C/min to reach the final temperature. Press ON to enable a temperature ramp.
Final temp 1	0–450 °C[1]	This parameter defines the temperature the column oven will reach at the end of the heating or cooling ramp. This line only appears if Ramp 1 is On.
Final time 1	0.00–999.99 min, ∞	This parameter defines how long (in minutes) the oven will maintain the final temperature of the ramp.
Ramp 2-7	On/Off, ∞ 0.0–120 °C/min	After you program the first ramp, the menu adds the Ramp 2 parameter lines. If you do not want an additional ramp, leave this parameter set to OFF. To program the ramp, press ON. The Final temp and Final time lines for the ramp will be added to the menu. You can repeat this process to program up to seven temperature ramps.
Final temp 2-7	0–450 °C[1]	This parameter defines the temperature the column oven will reach at the end of the relevant ramp.

Table 13-2. Oven Menu (Continued)

Menu	Range	Comments
`Final time 2-7`	0.00–999.99 min, ∞	This parameter defines how long (in minutes) the oven will maintain the final temperature of the ramp.
`Post run temp`	0–450 °C[1]	This parameter defines the temperature the oven will reach after the end of the analytical run. Press OFF if you do not want a post run temperature. Press ON to display the setpoint value and the `Post run temp`, `Post run time`, `L Post pres`, and `R post pres` parameters.
`Post run time`	0.00–999.99 min	This is the time the oven maintains the post run temperature.
`L/R Post pres`	0–700 kPa	This parameter defines the pressure for the Left or Right carrier during the `Post run time` when the system operates in constant pressure or programmed pressure mode.

1. With a cryogenic system, the ranges are –99 to -300 °C with liquid N_2 and –55 to –300 °C with liquid CO_2.

OPERATING SEQUENCE

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.

1. Press OVEN to open the **OVEN** menu.
2. Scroll to `Temp` and enter the initial temperature.
3. Scroll to `Initial time` and enter the time you want the oven to maintain the initial temperature.
4. Scroll to `Ramp 1` and press ON. Enter the ramp rate in °C/minute for the oven to reach the ramp's `Final temp`.
5. Scroll to `Final temp 1` and enter the final temperature for the ramp.
6. Scroll to `Final time 1` and enter the time the oven will maintain the `Final temp`.
7. To end the single ramp program, `Ramp 2` must be `Off`.

OPERATING SEQUENCE

Setting Up a Multiple Ramp Temperature Program

This program raises the initial oven temperature to a specified final temperature through up to seven ramps, each having a specified ramp rate, time, and temperature.

1. Press OVEN to open the **OVEN** menu.
2. Scroll to `Temp` and enter the initial temperature.
3. Scroll to `Initial time` and enter the time you want the oven to maintain the initial temperature.
4. Scroll to `Ramp 1` and press ON. Enter the ramp rate in °C/minute for the oven to reach the ramp's `Final temp`.
5. Scroll to `Final temp 1` and enter the final temperature for the first ramp.
6. Scroll to `Final time 1` and enter the time the oven will maintain the `Final temp`.
7. Scroll to `Ramp 2` and press ON. Enter the ramp rate for the second temperature ramp.
8. Scroll to `Final temp 2` and enter the final temperature for the second ramp.
9. Scroll to `Final time 2` and enter the time 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.

14

Columns

This chapter describes the analytical columns used in the TRACE GC Ultra. The operating sequences for leak test and column evaluation are also included.

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Connecting a Glass Packed Column to a TCD and to a S/SL injector 322
Performing a Leak Check 329
Performing a Column Evaluation 332

Introduction

The column is the heart of the gas chromatograph where the separation takes place. It is installed in the GC oven and connects the injector to the detector. The GC oven controller accurately controls the column temperature.

Each column has a maximum recommended operating temperature. To protect the column from excessively high temperatures, remember to set the `Max temp` parameter for the column oven in the **`CONFIGURE OVEN`** menu, as described in Chapter 13, *The Column Oven*.

Capillary and Wide-Bore Columns

The capillary and wide-bore capillary columns should be positioned inside the oven on the column support. The column ends should align correctly with the injector and detector base bodies. Wide-bore capillary columns can also be installed into the packed and purged packed injectors.

On-column injectors with autosamplers require a wide-bore pre-column. Pre-columns help prevent the *flooding effect* and prevent contamination of the analytical column. Refer to Chapter 7, *High Oven Temperature Cold On-Column Injector (HOT OC)*, for more information about pre-columns and using autosamplers with on-column injectors.

Using Correct Fittings

To connect a capillary column to the injector and detector base body, you must use the proper column ferrules and retaining nuts.

Column Ferrules

Graphite ferrules and graphitized Vespel® ferrules are used for many column connections.

- Encapsulated graphite ferrules connect the capillary column to the detector base body and to the S/SL and PTV injectors.
- Graphitized Vespel® ferrules are used *only* to connect capillary columns to on-column injectors.

CAUTION Overtightening compression ferrules does not necessarily produce a stronger, leak-free joint. In fact, very often the reverse is true. Too much pressure can cause a leak in the joint and make it very difficult to reseal that particular joint when changing columns.

Table 14-1 lists the ferrules to use depending on the pre-column and capillary column external diameter. Ferrules that are the wrong size cause leaks and contamination.

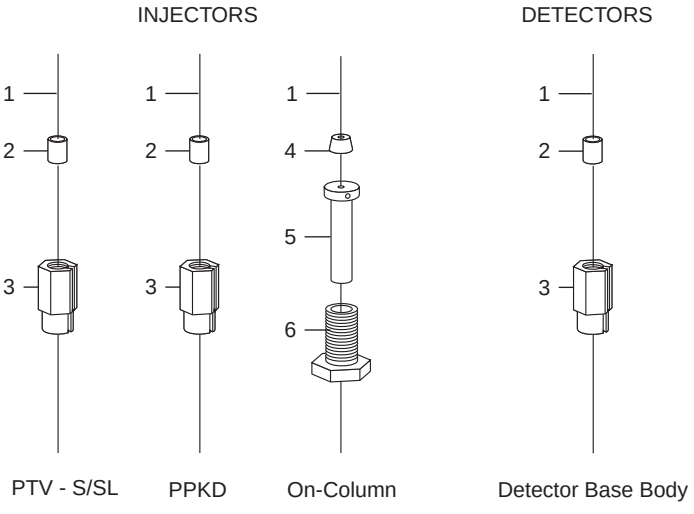
Table 14-1. Ferrules

Capillary Column	Graphite Ferrules	Graphitized Vespel® Ferrules
0.2 mm ID	0.25 mm ID	0.25 mm ID
0.25 mm ID	0.35 mm ID	0.35 mm ID
0.32 mm ID	0.45 mm ID	0.45 mm ID
0.53 mm ID	0.8 mm ID	0.8 mm ID

Retaining Nuts

M4 split retaining nuts are used to connect capillary columns to injector and detector base bodies. The nuts are split to allow easy installation and removal.

On-column injectors require a dedicated M8 retaining nut. Figure 14-1 shows how to connect capillary or wide column to injector and detector base body.



1. Capillary/Wide bore Column
2. Graphite Ferrule
3. M4 Split Retaining Nut
4. Graphitized Vespel® Ferrule
5. Back Washer/Sleeve for Secondary Cooling
6. M8 Retaining Nut
7. Front Ferrule
8. Back Ferrule
9. Nut

Figure 14-1. Capillary/Wide Bore Column to Injector and Detector Base Body Connections

Press-Fit Connections and Butt Connectors

Glass press-fit connectors couple the fused silica pre-column to the capillary column. Y press-fit connections are used for multi-detector configurations.

Figure 14-2 shows an example of press-fit connection.

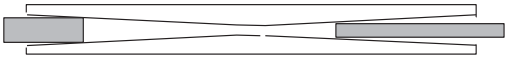
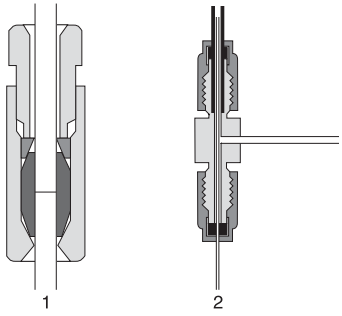


Figure 14-2. Press-Fit Connection

Figure 14-3 shows butt connectors for different applications.



1. Butt Connector Without Make-up Gas
2. Tee Butt Connector With Make-up Gas

Figure 14-3. Butt Connectors

- Figure 14-3 part 1 shows a butt connector with a single Vespel® or graphite ferrule used to connect a pre-column to an analytical column with the same diameter.
- Figure 14-3 part 2 shows a butt connector with make-up lines used to connect a pre-column, normally wide-bore, to an analytical column with a smaller diameter. The make-up line supplies a make-up gas to effectively flush the connection.

NOTE Press-fit connectors can be used instead of butt connectors in all cases.

OPERATING SEQUENCE

Installing the Column Support

To install the column support into the GC oven as, insert the four pins into the corresponding button-holes on the ceiling of the GC oven as shown in Figure 14-4.

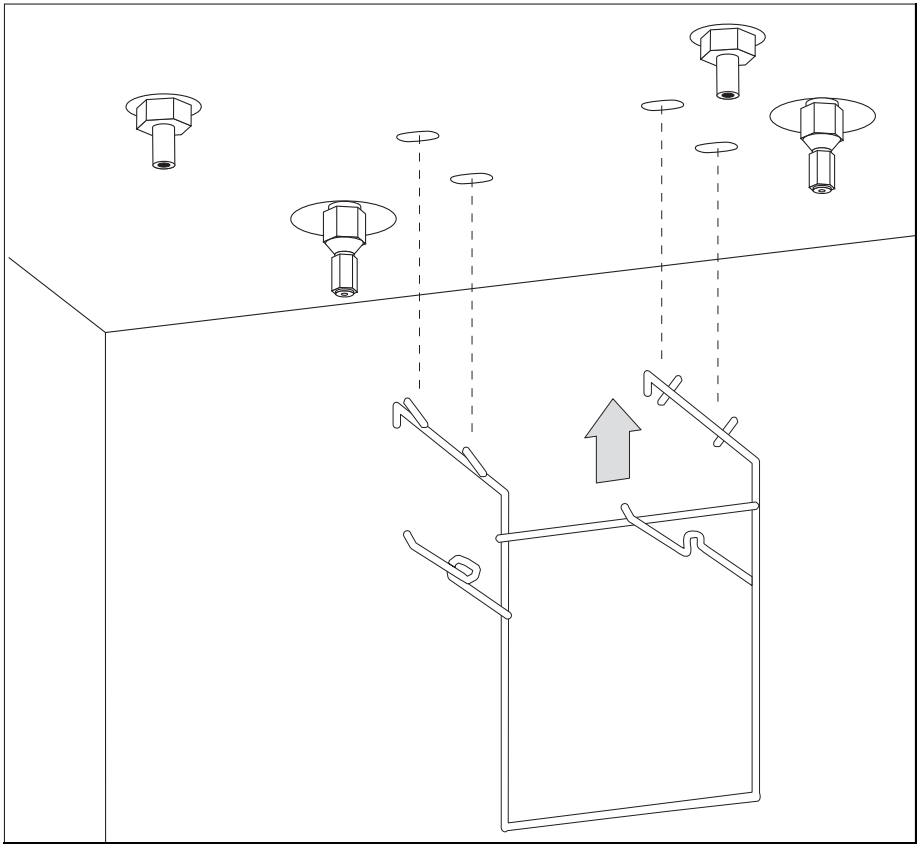


Figure 14-4. Column Support Installation

OPERATING SEQUENCE

Preparing a Capillary Column

To obtain a correct cut, use a ceramic scoring wafer (smooth edge) or sapphire scribe.

Materials required:

- ceramic scoring wafer or sapphire scribe

1. Hold the capillary column between your thumb and index finger with the column extending past the tip of your index finger.
2. Score the column very gently. Excessive force could crush the column end.
3. Snap off the end of the column.
4. Inspect the column end for an even, flat cut.

WARNING! Wear safety glasses to protect your eyes from flying particles while handling, cutting, or installing columns. Be careful handling columns to avoid accidental hand injuries.

PRECAUTIONS

OPERATING SEQUENCE

How to Use the Press-Fit Connectors

Glass press-fit connectors couple fused silica pre-column to a capillary column; e.g. to connect columns differing in polarity, or to repair a broken column.

For an optimum connections performance operate as follows:

1. Properly cut the fused silica column ends pay attention to achieve a clean square cut by using a ceramic scoring wafer or sapphire scribe.

CAUTION A poorly cut will produce an insufficient seal.

2. Insert the column ends into the relevant ports of the press-fit.

3. To create a good seal between all the parts, it is necessary to increase the oven temperature up to 200 °C.

OPERATING SEQUENCE

Connecting a Capillary Column to a S/SL Injector

Before connecting the column, make sure the injector has been properly assembled and programmed and the column support has been installed in the GC oven. For more information about split/splitless injectors, refer to Chapter 6, *On-Column Injector (OCI)*.

Materials required:

- M4 column retaining nut
- graphite ferrule
- typewriter correction fluid or a felt-tipped pen
- 6 mm wrench

1. Slide the graphite ferrule onto the capillary column with the bevelled end facing the injector. Be careful to avoid damaging the graphite ferrule when inserting the column.

2. Cut at least 1 cm from the column end. Refer to the *Preparing a Capillary Column* operating sequence on page 283 for instructions.

3. Place the column on the column support.

4. Use typewriter correction fluid or a felt-tipped pen to mark the correct position of the ferrule from the end of the column depending on the injection technique. The correct positions are as follows:

    - 40 mm for split injection
    - 64 mm for splitless injection
    - 50 mm for LVSL injection with packed liner
    - 60 mm for LVSL injection with laminar liner

5. Insert the column about 2 cm into the injector and slide the ferrule on the column up to the injector base, then slide the retaining nut onto the column through the side cut. The TRACE GC Ultra retaining nuts have a slotted design that makes them easy to add and remove.

6. Finger-tighten the column retaining nut until it starts to grip the column.

7. Adjust the column position so that the mark is even with the column retaining nut.

8. Use the 6 mm wrench to tighten the retaining nut using no more pressure than is necessary to obtain a good seal (1/4 to 1/2 turn).

9. Conduct a leak check of the column installation, as described in the *Packed Columns* operating sequence on page 305.

OPERATING SEQUENCE

Connecting a Capillary Column to an OC Injector

Before you begin this sequence, insert the syringe needle into the injector. If you are using a pre-column, connect it to the capillary column using a press-fit or butt connector. Also make sure the column support has been installed in the GC oven. For more information about on-column injectors, refer to Chapter 7, *High Oven Temperature Cold On-Column Injector (HOT OC)*.

Materials required:

- M8 retaining nut
- backwasher/sleeve for secondary cooling
- graphitized Vespel® ferrule
- 10 mm wrench

1. Slide the M8 Vespel® ferrule, the secondary cooling sleeve, and the retaining nut onto the capillary column (or pre-column, if used). See Figure 14-1 on page 280 for the correct assembly order.

NOTE If the HOT device is used, the M8 retaining nut and HOT device are used in place of the standard secondary cooling sleeve.

2. Slide the column onto the needle protruding into the column oven, then push the column into the injector as far as it will go.

3. Place the column on the column support.

4. Slide the ferrule, the retaining nut, and the secondary cooling sleeve onto the column and tighten the nut onto the injector with a 10 mm wrench until the column is secure. Use no more pressure than is necessary to ensure a good seal.

5. Remove the syringe needle and reinsert it. It should slide easily into the column without friction. If not, repeat the column installation sequence.

To check that the column positioning in the on-column injector has not blocked the carrier gas path, turn on the carrier gas line. You should hear carrier gas escaping through the syringe needle channel when the injection valve is opened.

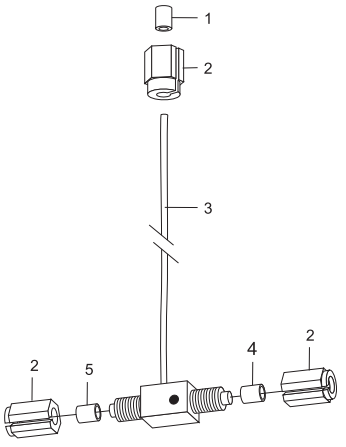
6. Leak check the column, as described in the *Performing a Leak Check* operating sequence on page 329.

OPERATING SEQUENCE

Connecting the Large Volume Injection System Tee Piece

Materials required:

- Uncoret™ 12 m, 0.53 mm ID uncoated pre-column as a retention gap with 3 m long coated segment (SE-54; 0.45 µm film thickness)
- 0.32 or 0.25 mm ID fused silica capillary column
- tee connector with M4 column retaining nuts and graphite ferrules, as shown in Figure 14-5
- 7 mm wrench
- 10 mm wrench



1. 1 mm OD Graphitized Vespel Ferrule
2. M4 Retaining Nut
3 SVE Line Metal Tube
4. 0.8 mm Graphite Ferrule for 0.53 mm ID Column
.5. 0.45 mm Graphite Ferrule for 0.32 mm ID Column

Figure 14-5. Tee Connection Assembly

NOTE Before starting, insert the AS autosampler syringe needle into the injector.

We recommend that you connect the analytical column to the detector *after* a leak test of the Solvent Vapor Exit (SVE) system.

Connect the Uncoret™ Pre-Column

1. Connect the Uncoret™ pre-column to the on-column injector as described in the *Connecting a Capillary Column to an OC Injector* operating sequence on page 287.
2. Slide the 0.8 mm graphite ferrule onto the pre-column with the bevelled end facing the tee connector. Be careful to avoid damaging the graphite ferrule when inserting the column.
3. Cut 1 cm from the pre-column end.
4. Insert the pre-column into the tee connector.
5. Slide the M4 retaining nut on the column through the side cut.
6. Tighten the column retaining nut until it starts to grip the pre-column.

Connect the Analytical Column

1. Slide the 0.45 mm graphite ferrule onto the column with the bevelled end facing the tee piece. Be careful to avoid damaging the graphite ferrule when inserting the column.
2. Cut 1 cm from the column end. Refer to the *Preparing a Capillary Column* operating sequence on page 283 for more instructions.
3. Place the column on the column support.
4. Insert the analytical column end through the tee connector as shown in Figure 14-6.
5. Slide the M4 retaining nut onto the column through its side cut.
6. Finger-tighten the column retaining nut until it starts to grip the column.

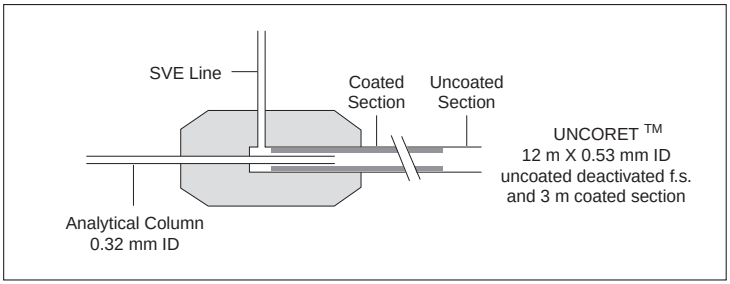


Figure 14-6. Uncoret™ Pre-Column/Column Connection

Connect the SVE system

1. On the GC injector/detector cassette, unscrew the SVE valve fixing screws then lift the valve from its seat. In the GC column oven, guide the SVE line metal tube through the oven ceiling and the injector/detector cassette until it protrudes the SVE system.

2. Slide the M4 retaining nut then the 1 mm OD graphitized Vespel ferrule onto the SVE line metal tube. The bevelled open end should face the SVE system. Be careful to avoid damaging the ferrule.

3. Insert the SVE line metal tube into the SVE valve inlet.

4. Finger-tighten the metal tube retaining nut until it starts to grip the SVE valve inlet.

5. Use the 6 mm wrench to tighten the retaining nuts. Use no more pressure than is necessary to obtain a good seal (1/4 to 1/2 turn).

6. Place the SVE valve in the its proper seat. Fix the valve by using the two fixing screws.

The result of this operation is shown in Figure 14-7.

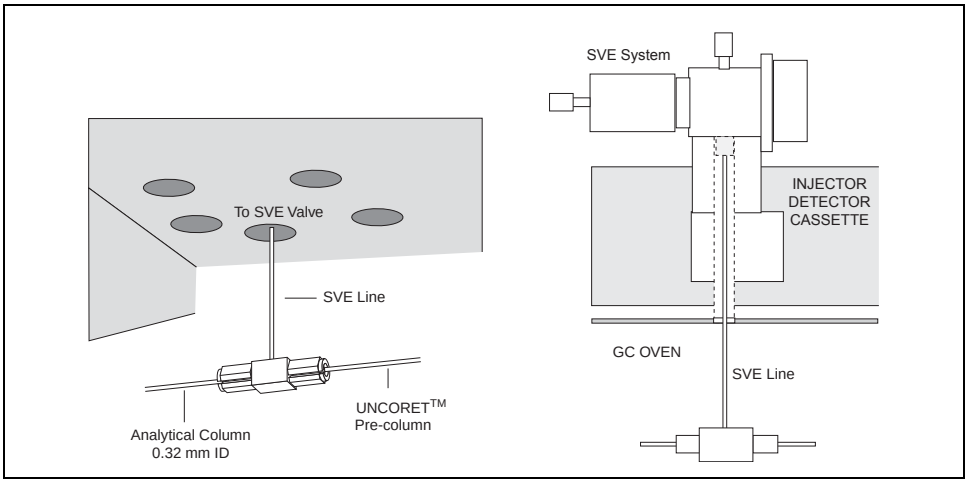


Figure 14-7. Large Volume Injection Tee Connection

7. Leak check the column, as described in the *Performing a Leak Check* operating sequence on page 329.

OPERATING SEQUENCE

Connecting a Wide-Bore Column to a PPKD Injector

Before you begin, make sure the column support has been installed in the GC oven.

Materials required:

- M4 column retaining nut
- graphite ferrule
- 6 mm wrench

1. Slide the graphite ferrule onto the wide-bore column with the bevelled end facing the injector. Be careful to avoid damaging the graphite ferrule when inserting the column.
2. Cut 1 cm from the column end. Refer to the *Preparing a Capillary Column* operating sequence on page 283 for instructions.
3. Place the column on the column support.
4. Insert the column into the injector and slide the ferrule up to the injector base.
5. Slide the M4 retaining nut onto the column through its side cut.
6. Finger-tighten the column retaining nut until it starts to grip the column.
7. Adjust the column position so that its end rests against the bottom of the liner.
8. Use the 6 mm wrench to tighten the retaining nut. Use no more pressure than is necessary to obtain a good seal (1/4 to 1/2 turn).
9. Conduct a leak check of the column installation, as described in the *Packed Columns* operating sequence on page 305.

OPERATING SEQUENCE

Connecting a Capillary Column to a PTV Injector

Before you begin, make sure the column support has been installed in the GC oven (page 282).

Materials required:

- M4 column retaining split nut
- Graphite ferrule
- 6 mm wrench

1. Slide the graphite ferrule onto the wide-bore column with the bevelled end facing the injector. Be careful to avoid damaging the graphite ferrule when inserting the column.
2. Cut 1 cm from the column end. Refer to the *Preparing a Capillary Column* operating sequence on page 283 for instructions.
3. Place the column on the column support.
4. Insert the column into the injector and slide the ferrule up to the injector base.
5. Insert the column about 30 mm into the bottom of the injector.
6. Slide the M4 retaining nut onto the column through its side cut.
7. Finger-tighten the column retaining nut until it starts to grip the column.
8. Use the 6 mm wrench to tighten the retaining nut. Use no more pressure than is necessary to obtain a good seal (1/4 to 1/2 turn).
9. Conduct a leak check of the column installation, as described in the *Performing a Leak Check* operating sequence on page 329.

Using the PTV for On-Column Injections

Use the following column installation sequence if you wish to use the PTV for injections similar to on-column injections:

1. Slide the graphite ferrule onto the wide-bore column with the bevelled end facing the injector. Be careful to avoid damaging the graphite ferrule when inserting the column.
2. Cut 1 cm from the column end. Refer to the *Preparing a Capillary Column* operating sequence on page 283 for instructions.
3. Place the column on the column support.
4. Insert the column into the injector and slide the ferrule up to the injector base.
5. Insert the column as far as possible into the bottom of the injector.
6. Slide the M4 retaining nut onto the column through its side cut.
7. Finger-tighten the column retaining nut until it starts to grip the column.
8. Use the 6 mm wrench to tighten the retaining nut. Use no more pressure than is necessary to obtain a good seal (1/4 to 1/2 turn).
9. Conduct a leak check of the column installation, as described in the *Performing a Leak Check* operating sequence on page 329.

OPERATING SEQUENCE

Connecting a Capillary Column to an FID, NPD, or FPD

Before beginning this sequence, remove the detector from the detector base body.

Materials required:

- M4 column retaining nut
- graphite ferrule
- 6 mm wrench

1. Slide the graphite ferrule onto the capillary column with the bevelled end facing the detector base body. Be careful to avoid damaging the graphite ferrule when inserting the column.
2. Cut 2–3 cm from the column end. Refer to the *Preparing a Capillary Column* operating sequence on page 283 for instructions.
3. Insert the column into the detector base body and slide the ferrule up to the detector base body.
4. Slide the M4 retaining nut onto the column through its side cut.
5. Finger-tighten the column retaining nut until it starts to grip the column.
6. Push the column through the detector base body and into the jet. Depending on the column dimensions, the column may pass through the jet. Pull the column back so that the end of the column is 2 to 3 mm below the tip of the jet. The column insertion depths measured from the bottom of the ferrule are 94 mm for FID, 97 mm for NPD and 127 mm for FPD.
7. Use the 6 mm wrench to tighten the retaining nut. Use no more pressure than is necessary to obtain a good seal (1/4 to 1/2 turn).

WARNING! Be especially careful when using a metal column. With the detector in place, the tip of the jet is polarized to high voltage. The metal column must never touch the tip of the jet. Contact of the metal column with the electrically charged tip can cause electrical shock and damage to the instrument.

OPERATING SEQUENCE

Connecting a Capillary Column to an ECD

Before beginning this sequence, remove the detector from the detector base body.

Materials required:

- M4 column retaining nut
- graphite ferrule
- 6 mm wrench

1. Slide the graphite ferrule onto the capillary column with the bevelled end facing the detector base body. Be careful to avoid damaging the graphite ferrule when inserting the column.
2. Cut 2–3 cm from the column end. Refer to the *Preparing a Capillary Column* operating sequence on page 283 for instructions.
3. Insert the column into the detector base body and slide the ferrule up to the detector base body.
4. Slide the M4 retaining nut onto the column through its side cut.
5. Finger-tighten the column retaining nut until it starts to grip the column.
6. Adjust the column position so that it protrudes about 2 cm above the top of the detector base body (109 mm for the bottom of the ferrule).
7. Use the 6 mm wrench to tighten the retaining nut. Use no more pressure than is necessary to obtain a good seal (1/4 to 1/2 turn).

OPERATING SEQUENCE

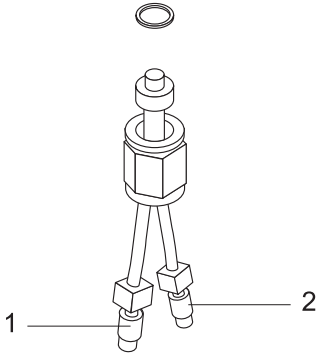
Connecting a Capillary Column to a PID

Materials required:

- two-way capillary adapter
- two M4 split retaining nuts
- graphite ferrule
- exit line (700 mm long, 0.53 mm ID deactivated uncoated fused silica tubing)
- 0.8 mm ID graphite ferrule for the exit line
- typewriter correction fluid, or felt-tipped pen
- 10 mm wrench
- 6 mm wrench
- 5 mm wrench

Connect the column to the detector inlet

1. Attach the two-way capillary column adapter (shown in Figure 14-8) to the lower end of the detector base body and tighten it by using 10 mm wrench.



1. Column Inlet 2. Exit Line

Figure 14-8. Two-Way Capillary Adapter

2. Leak test the detector. Refer to *TRACE GC Ultra Maintenance and Troubleshooting Manual.*

3. Slide the graphite ferrule onto the capillary column with the bevelled end facing the detector base body. Be careful to avoid damaging the graphite ferrule when inserting the column.

4. Cut 2–3 cm from the column end. Refer to the *Preparing a Capillary Column* operating sequence on page 283 for instructions.

5. Use the typewriter correction fluid or a felt-tipped pen to mark the column 132–135 mm from the column end (12–15 mm from the upper end of the detector base body).

6. Gently insert the column into one of the two inlet ports of the two-way capillary adapter. Use the mark as a guide to determine how far to insert the column.

7. Slide the M4 retaining nut onto the column through its side cut.

8. Finger-tighten the column retaining nut until it starts to grip the column.

9. Adjust the column position so that the mark is even with the column retaining nut.

10. Using the 5 mm wrench, keep blocked the inlet adapter nut then use the 6 mm wrench to tighten the retaining nut. Use no more pressure than is necessary to obtain a good seal (1/4 to 1/2 turn).

If correctly positioned, the column enters the detector cell block 12–15 mm from the upper end of the detector base body.

Connect the Exit Line

The exit line must always be connected, even when you do not wish to have a second detector coupled in series with the PID. In this case, an outlet end of the exit line should either be connected to an unused detector base body or exit from the GC oven passing through one of the holes in the oven ceiling, as shown in Figure 13-1 on page 264.

1. Slide the 0.8 mm ID graphite ferrule onto the uncoated fused silica column with the bevelled end facing the detector base body or exit. Be careful to avoid damaging the graphite ferrule when inserting the column.

2. Cut 2–3 cm from the column end. Refer to the *Preparing a Capillary Column* operating sequence on page 283 for instructions.

3. Use the typewriter correction fluid or a felt-tipped pen to mark the column 143–145 mm (23–25 mm for the CB 70 detector base body) from the column end.

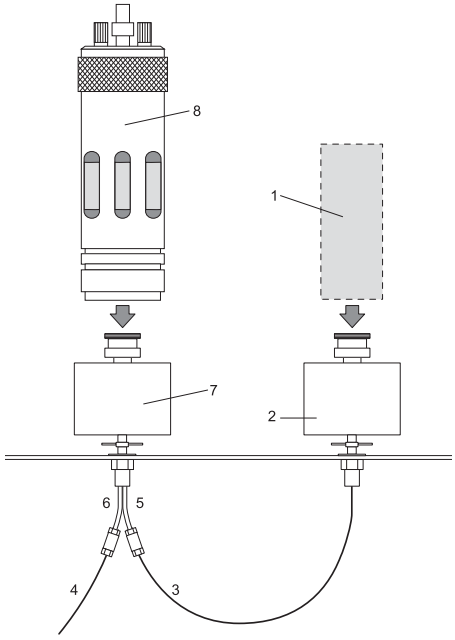
4. Gently insert the column into the second inlet port of the two-way adapter. Use the mark as a guide to determine how far to insert the column. If correctly positioned, the exit line enters the detector cell block 23–25 mm from the upper end of the detector base body.

5. Slide the M4 retaining nut onto the column through its side cut.

6. Finger-tighten the column retaining nut until it starts to grip the column.

7. Use the appropriate wrench to tighten the retaining nuts. Use no more pressure than is necessary to obtain a good seal (1/4 to 1/2 turn).

Figure 14-9 shows the column connections for the PID and a detector coupled with an auxiliary detector.



1. Auxiliary Detector
2. 2nd Detector Base Body
3. Fused Silica Tubing
4. Column
5. Exit Line
6. Inlet Line
7. Detector Base Body
8. PID

Figure 14-9. PID Column Connections

OPERATING SEQUENCE

Connecting a Capillary Column to a TCD

Before you connect the capillary column to the TCD, be sure to do the following:

- condition the column
- make sure the capillary column adapter is mounted on the detector base body

Materials required:

- M4 column retaining nut
- graphite ferrule
- 6 mm wrench
- capillary column adapter

1. Connect the capillary column adapter to the detector base body.
2. Slide the graphite ferrule onto the column with the beveled end facing the injector. Be careful to avoid damaging the graphite when inserting the column.
3. Cut 2–3 cm from the column end. Refer to the *Preparing a Capillary Column* operating sequence on page 283 for instructions.
4. Insert the column into the detector base body and slide the ferrule up to the detector base body.
5. Slide the M4 retaining nut onto the column through its side cut.
6. Finger-tighten the retaining nut until it starts to grip the column.
7. Push the column all the way up into the detector, then pull the column out about 1 mm.
8. Tighten the M4 retaining nut using the 6 mm wrench. Use no more pressure than is necessary to achieve a good seal (1/4 to 1/2 turn).

OPERATING SEQUENCE

Connecting a Capillary Column to an PDD

Before beginning this sequence, remove the detector from the detector base body.

Materials required:

- M4 column retaining nut
- graphite ferrule
- 6 mm wrench

1. Slide the graphite ferrule onto the capillary column with the bevelled end facing the detector base body. Be careful to avoid damaging the graphite ferrule when inserting the column.
2. Cut 2–3 cm from the column end. Refer to the *Preparing a Capillary Column* operating sequence on page 283 for instructions.
3. Insert the column into the detector base body and slide the ferrule up to the detector base body.
4. Slide the M4 retaining nut onto the column through its side cut.
5. Finger-tighten the column retaining nut until it starts to grip the column.
6. Adjust the column position so that it protrudes about 33-35 mm above the top of the detector base body (123 mm for the bottom of the ferrule).
7. Use the 6 mm wrench to tighten the retaining nut. Use no more pressure than is necessary to obtain a good seal (1/4 to 1/2 turn).

Column Insertion Depths Summary Tables

The following Tables 14-2 and 14-3 reassume the injectors and detectors column insertion depths measured from the bottom of the ferrule.

Table 14-2. Column Insertion Depths for Injectors

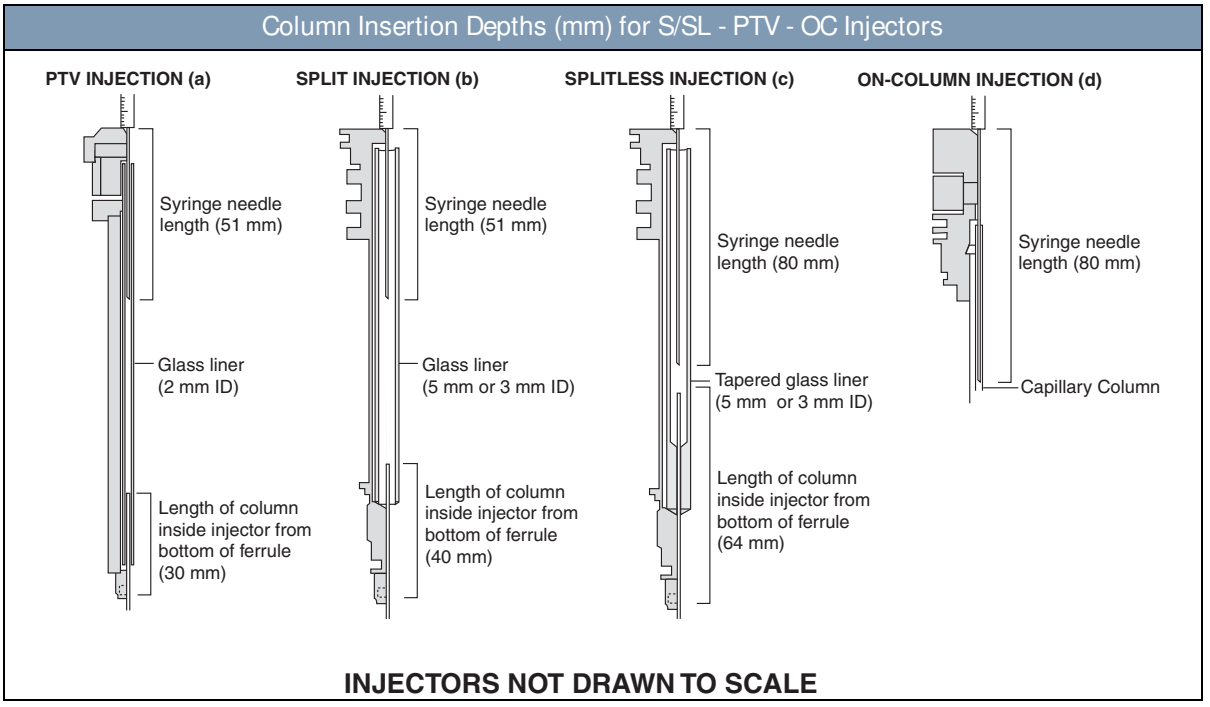
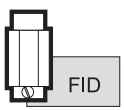
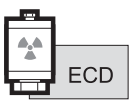
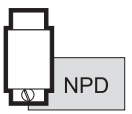
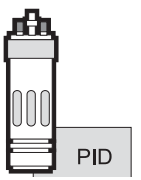
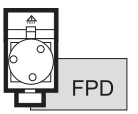
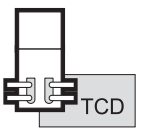
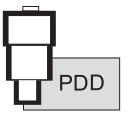


Table 14-3. Column Insertion Depths for Detectors

Column Insertion Depths (mm) for FID - ECD - NPD - PID - FPD - TCD - PDD Detectors						
						
94	109	97	135 Column 144 Exit L.	127	As far as it will go	123

Packed Columns

There are different sizes of packed columns with both metric and imperial dimensions with dedicated adapters. The TRACE GC Ultra accepts 1/4 inch OD, 1/8 inch OD imperial metal packed columns, 6 mm OD metric metal packed columns and 6 mm OD glass packed columns. With the appropriate conversion kit, you can also install metal packed columns into the S/SL injector.

Metric Packed Columns

The following metric packed columns are commonly used:

- 6 mm OD packed metal column
- 6 mm OD packed glass column

Using Correct Metric Fittings

To connect packed columns to injector and detector base bodies, you must use the correct column ferrules and retaining nuts.

Metric Column Ferrules

The ferrule size should be compatible with the packed column. The type of ferrule you use depends on the type of packed column:

- metal packed columns require double brass ferrules (front and back)
- glass packed columns require Viton® O-ring or PTFE ferrules

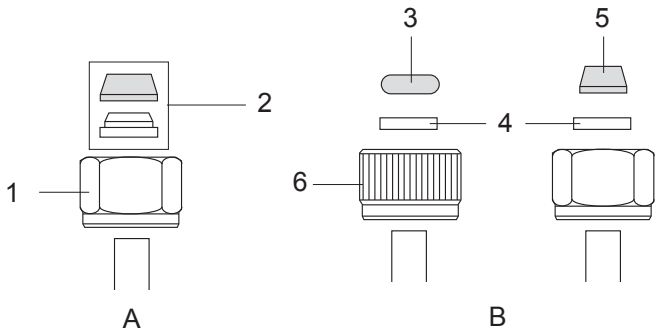
Metric Retaining Nuts

Use hexagonal 1/4 inch column retaining nuts to connect all metal packed columns and round 1/4 inch knurled or grooved nuts to connect glass packed columns.

Table 14-4 lists the correct fittings for different sizes of metric packed columns. Figure 14-10 shows the fittings.

Table 14-4. Metric Packed Column Fittings

Column Type	Ferrules	Retaining Nut
6 mm OD metal column	brass double	hexagonal 1/4 inch
6 mm OD glass column	Viton® O-Ring + washer	round 1/4 inch



1. Hexagonal 1/4 Inch Nut
2. Double Brass Ferrules
3. Viton® O-Ring
4. Washer
5. PTFE Ferrule
6. Round 1/4 Inch Knurled Nut

A. 6 mm OD Metal Column
B. 6 mm OD Glass Column

Figure 14-10. Metric Packed Column Fittings

Imperial Packed Columns

The following types of imperial packed columns are commonly used.

- 1/4 inch OD metal packed column
- 1/8 inch OD metal packed column

Using Correct Imperial Fittings

To connect packed columns to injector and detector base bodies, you must use the correct column ferrules and retaining nuts.

Imperial Column Ferrules

The ferrule size should be compatible with the packed column.

- Use Swagelok® ferrules (front and back) with a 1/4 inch hexagonal nut to connect 1/4 inch metal packed columns to injector and detector metric/ imperial adapters.
- Use Swagelok® ferrules (front and back), and Swagelok® nuts to connect 1/8 inch metal packed columns to injector and detector metric/imperial adapters.

Imperial Retaining Nuts

Use Swagelok® nuts to connect all packed columns.

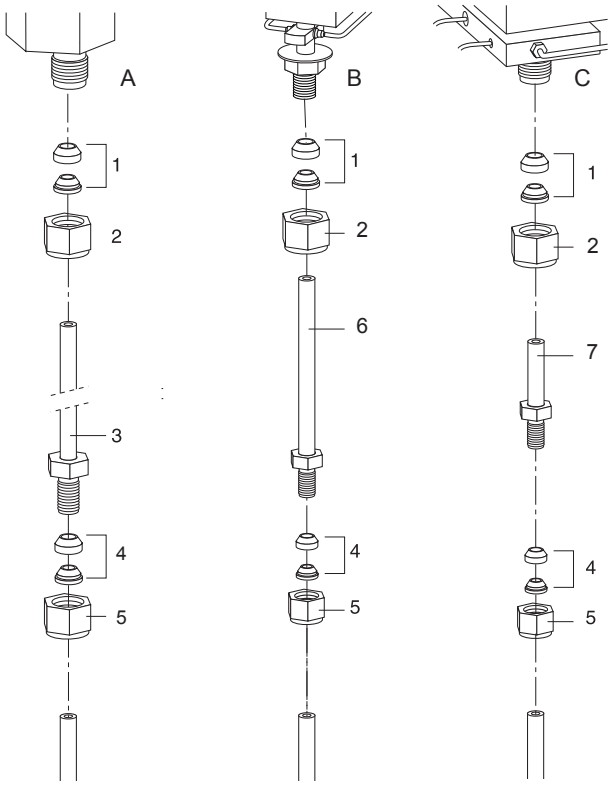
Table 14-5 lists the correct fittings depending on the type of imperial packed column.

Table 14-5. Imperial Size Packed Column Fittings

Column Type	Ferrules	Retaining Nut
metal column 1/4 inch	Swagelok® 1/4 inch	hexagonal 1/4 inch
metal column 1/8 inch	Swagelok® 1/8 inch	Swagelok® 1/8 inch

Adapters for Metal Packed Columns

To connect metal packed columns to the PKD or PPKD injector and the detector base bodies, you must use a proper metal metric/imperial adapter. Figure 14-11 shows an example of adapters.



A. PKD or PPKD Injector	3. Adapter for PKD or PPKD Injector
B. Detector Base Body for Packed Column	4. Double Ferrule
C. TCD Base Body	5. Nut
1. Double Ferrule	6. Adapter for Detector Base Body
2. Nut	7. Adapter for TCD

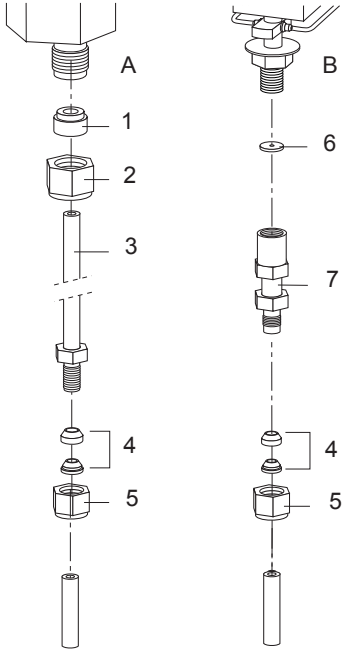
Figure 14-11. Injector and Detector Base Body Adapters

The adapters size depends on the type of:

- column that has to be use: 6-mm, 1/4-inch, 1/8-inch OD
- injector installed on the GC: PKD, PPKD
- detector base body installed on the GC: for packed columns, for TCD

NOTE

Metal Packed Column may be installed into the S/SL injector and the detector base body for capillary column by using the appropriate conversion kit as shown in Figure 14-12.



A. S/SL Injector	4. Double Ferrule
B. Detector Base Body for Capillary Column	5. Nut
1. Ferrule	6. Ferrule
2. Nut	7. Adapter for Detector Base Body
3. Adapter for S/SL Injector	

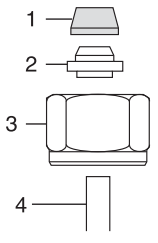
Figure 14-12. Conversion Kit

OPERATING SEQUENCE

Preparing a Metal Packed Column

Before you begin, verify that the proper adapters are installed on the injector and detector side.

Slide the fittings onto the packed column injector and detector ends in the order and direction shown in Figure 14-13.



1. Front Ferrule	3. Retaining Nut
2. Back Ferrule	4. Packed Column

Figure 14-13. Metal Packed Column Fittings

OPERATING SEQUENCE

Connecting a Metal Packed Column to a PKD or PPKD Injector

Materials required:

- retaining nut
- ferrules
- 10 mm or 1/4 inch wrench
- adapter for injector

1. Make sure that your packed column has been correctly prepared as described in the *Preparing a Metal Packed Column* operating sequence on page 310.
2. Insert the appropriate adapter into the bottom of the injector, then push up the adapter into the injector as far as possible.
3. Slide the ferrule up to injector base then finger-tighten the adapter retaining nut until it starts to grip the adapter.
4. Use the wrench to tighten the retaining nut. Use no more pressure than is necessary to obtain a good seal (1/4 to 1/2 turn).
5. Insert the inlet end of the column to the adapter base as far as possible.
6. Slide the ferrule up to adapter base then finger-tighten the column retaining nut until it starts to grip the column.
7. Use the wrench to tighten the retaining nut. Use no more pressure than is necessary to obtain a good seal (1/4 to 1/2 turn).

CAUTION Overtightening the compression ferrule does not necessarily produce a stronger, leak-free joint. In fact, very often the reverse is true. Overtightening can cause a leak in the joint and make it very difficult to reseal that particular joint when changing columns.

OPERATING SEQUENCE

Connecting a Metal Packed Column to an FID, NPD, FPD, or ECD

Materials required:

- retaining nut
- ferrules
- 10 mm or 1/4 inch wrench
- adapter for detector

1. Make sure that your packed column has been correctly prepared as described in the *Preparing a Metal Packed Column* operating sequence on page 310.
2. Insert the appropriate adapter into the bottom of the detector base, then push up the adapter into the detector base as far as possible.
3. Slide the ferrule up to detector base then finger-tighten the adapter retaining nut until it starts to grip the adapter.
4. Use the wrench to tighten the retaining nut. Use no more pressure than is necessary to obtain a good seal (1/4 to 1/2 turn).
5. Insert the detector end of the column to the adapter base as far as possible.
6. Slide the ferrule up to adapter base then finger-tighten the column retaining nut until it starts to grip the column.
7. Use the wrench to tighten the retaining nut. Use no more pressure than is necessary to obtain a good seal (1/4 to 1/2 turn).

CAUTION Overtightening the compression ferrule does not necessarily produce a stronger, leak-free joint. In fact, very often the reverse is true. Overtightening can cause a leak in the joint and make it very difficult to reseal that particular joint when changing columns.

OPERATING SEQUENCE

Connecting a Metal Packed Column to a TCD

Materials required:

- metric/imperial retaining nut
- metric/imperial ferrules
- 10 mm or 1/4 inch wrench
- adapter for detector

1. Insert the appropriate adapter into the bottom of the detector base, then push up the adapter into the detector base as far as possible.
2. Slide the ferrule up to detector base then finger-tighten the adapter retaining nut until it starts to grip the adapter.
3. Use the wrench to tighten the retaining nut. Use no more pressure than is necessary to obtain a good seal (1/4 to 1/2 turn).
4. Insert the detector end of the column to the adapter base as far as possible.
5. Slide the ferrule up to adapter base then finger-tighten the column retaining nut until it starts to grip the column.
6. Use the wrench to tighten the retaining nut. Use no more pressure than is necessary to obtain a good seal (1/4 to 1/2 turn).

CAUTION Overtightening the compression ferrule does not necessarily produce a stronger, leak-free joint. In fact, very often the reverse is true. Overtightening can cause a leak in the joint and make it very difficult to reseal that particular joint when changing columns.

OPERATING SEQUENCE

Connecting a Metal Packed Column to an PDD

Before beginning this sequence remove the detector from the detector base body. During this operation make care to withdraw the detector vertically.

NOTE

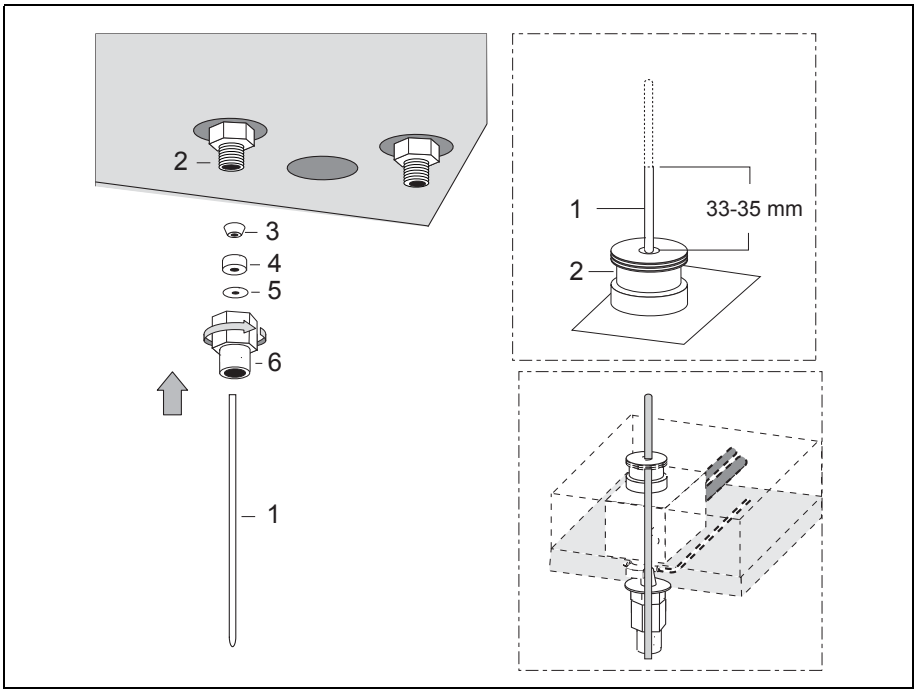
The PDD is compatible ONLY with 1/8-inch OD packed columns (NO 1/4-inch SS or 6 mm OD glass columns)

Materials required:

- PDD fixing tool
- graphitized Vespel® ferrule
- washer
- silver seal
- 0.7 mm ID fused silica capillary tubing (about 30 cm length)
- adapter for detector
- 6MB-1/8 inch Swagelock® column adapter
- retaining nut
- ferrules
- 8 mm wrench
- 10 mm or 1/4 inch wrench

1. Make sure that your packed column has been correctly prepared as described in the *Preparing a Metal Packed Column* operating sequence on page 310.

2. Cut about 30 cm of capillary tubing.
   Insert the appropriate fittings on the capillary tubing in the following order as shown in Figure 14-14.
   - adapter for detector
   - silver seal
   - washer
   - graphitized Vespel® ferrule



1. 0.7 mm ID Fused Silica Capillary Tubing	2. Detector Base Body
3. Graphitized Vespel® ferrule	4. Washer
5. Silver Seal	6. Adapter for Detector.

Figure 14-14. Connection to the PDD Detector (1)

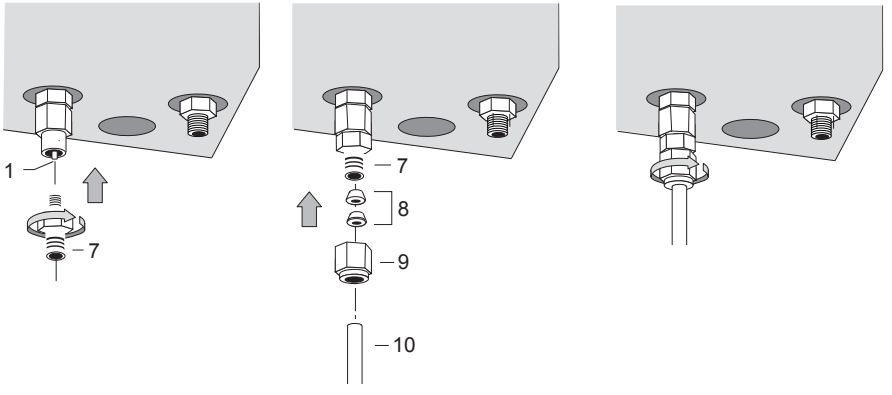
3. Insert the capillary tubing and its fittings into the bottom of the detector base.

4. Push up the capillary tubing until the upper end protrudes from the detector base body and the lower end is at the same level of the base of the adapter retaining nut as shown in Figure 14-14.
   Then push up the capillary tubing by screwing up a blind 6MB nut into the adapter in order to maintain the lower of the capillary completely inside the adapter.

5. Finger-tighten the adapter retaining nut until it starts to grip the detector base. The capillary tubing must protrude from the detector base body from 33-35 mm, then cut the exceeding portion as shown in Figure 14-14.

6. Use the wrench to tighten the retaining nut. Use no more pressure than is necessary to obtain a good seal (1/4 to 1/2 turn).

7. Insert the column adapter into the base of the adapter for detector as shown in Figure 14-15.



8. Double Ferrule	7. Column Adapter
10. Packed Column	9. Column Nut

Figure 14-15. Connection to the PDD Detector (2)

8. Finger-tighten the column adapter retaining nut until it starts to grip the adapter for detector.

9. Use the wrench to tighten the retaining nut. Use no more pressure than is necessary to obtain a good seal (1/4 to 1/2 turn).

10. Insert the detector end of the column to the column adapter base.

11. Slide the ferrule up to adapter base then finger-tighten the column retaining nut until it starts to grip the column.

12. Use the wrench to tighten the retaining nut. Use no more pressure than is necessary to obtain a good seal (1/4 to 1/2 turn).

CAUTION Overtightening the compression ferrule does not necessarily produce a stronger, leak-free joint. In fact, very often the reverse is true. Overtightening can cause a leak in the joint and make it very difficult to reseal that particular joint when changing columns.

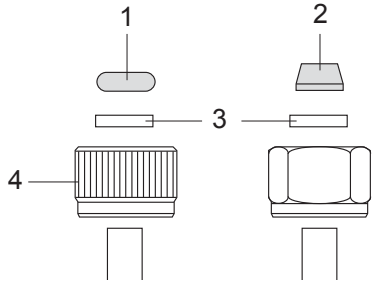
OPERATING SEQUENCE

Preparing a Glass Packed Column

Before you begin, verify that the injector and the detector base bodies are compatible with your metric or imperial column. Install the proper adapters if you are installing an imperial packed column.

NOTE Packed columns have one end longer than the other. Usually the longer end connects to the detector base body and the shorter end connects to the injector. Depending on the application, the connection may be reversed.

Slide the fittings onto the packed column injector and detector ends in the order and direction shown in Figure 14-16.



1. Viton® O-Ring
2. PTFE Ferrule
3. Washer
4. Knurled Nut

Figure 14-16. Glass Packed Column Fittings

OPERATING SEQUENCE

Connecting a Glass Packed Column to a TCD and to a PKD or PPKD injector

Materials required:

- retaining nut
- ferrules
- 60 mm glass liner

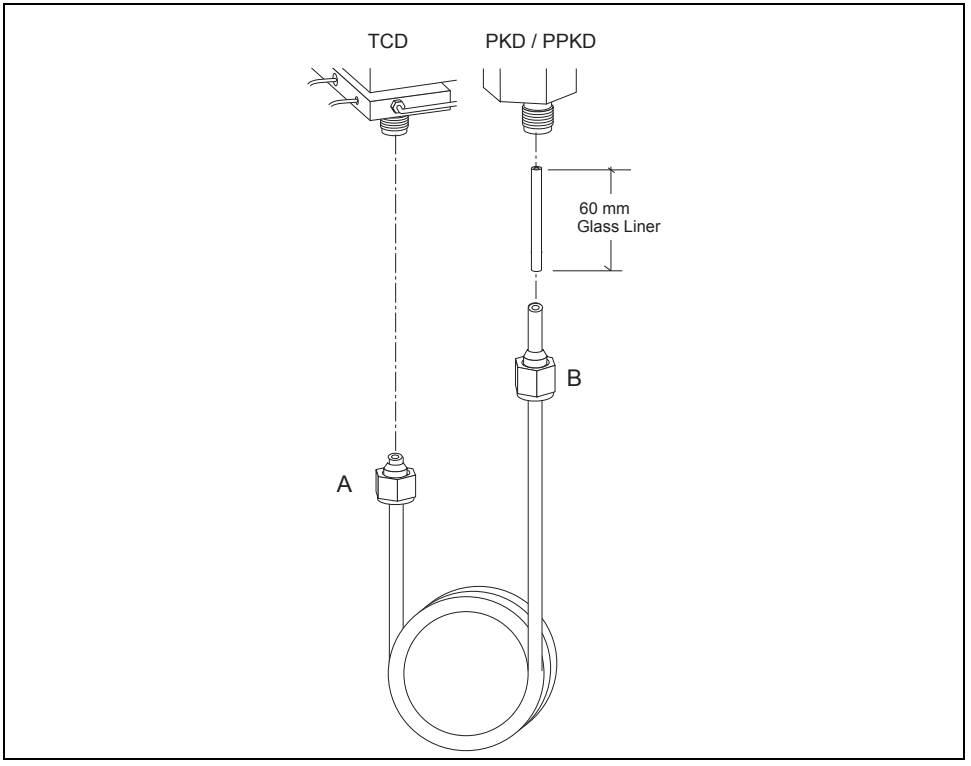


Figure 14-17. TCD-PKD/PPKD Configuration

In this configuration the longer end of the column connects to the injector and the shorter end connects to the detector base as shown in Figure 14-17. The use of a 60 mm glass liner is required.

1. Make sure that your packed column has been correctly prepared as described in the *Preparing a Glass Packed Column* operating procedure on page 317.

2. Insert the liner into the bottom of the injector.

3. Simultaneously insert the column ends A and B respectively into the detector and injector bodies paying attention that:

   — the column end A touches the bottom of the detector base.

   — the liner and the column end B are pushed up into the injector as far as possible.

4. Finger-tighten the column ends A and B retaining nuts until they start to grip the column.

CAUTION Overtightening the compression ferrule does not necessarily produce a stronger, leak-free joint. In fact, very often the reverse is true. Overtightening can cause a leak in the joint and make it very difficult to reseal that particular joint when changing columns.

OPERATING SEQUENCE

Connecting a Glass Packed Column to an FID, NPD, FPD or ECD and to a PKD or PPKD injector

Materials required:

- retaining nut
- ferrules
- 100 mm glass liner

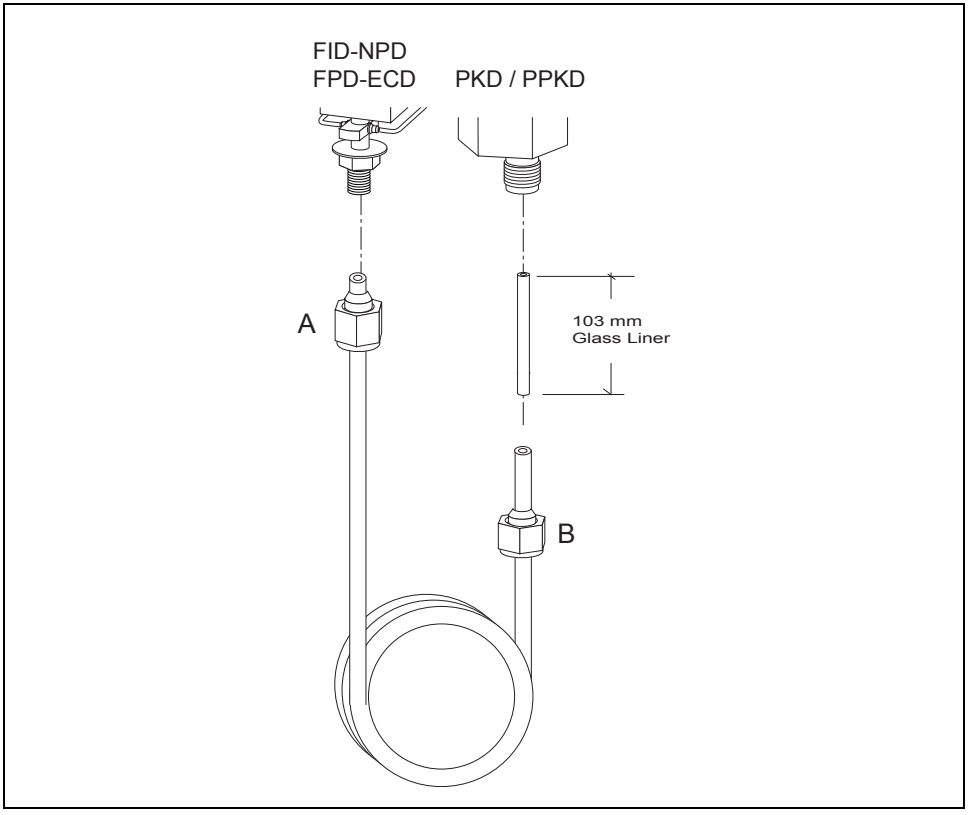


Figure 14-18. FID,NPD,FPD,ECD-PKD/PPKD Configuration

In this configuration the longer end of the column connects to the detector base body and the shorter end connects to the injector as shown in Figure 14-17. The use of a 100 mm glass liner is required.

1. Make sure that your packed column has been correctly prepared as described in the *Preparing a Glass Packed Column* operating procedure on page 317.

2. Insert the liner into the bottom of the injector.

3. Simultaneously insert the column ends A and B respectively into the detector and injector bodies paying attention that:

   — the column end A touches the bottom of the detector base.

   — the liner and the column end B are pushed up into the injector as far as possible.

4. Finger-tighten the column ends A and B retaining nuts until they start to grip the column.

CAUTION Overtightening the compression ferrule does not necessarily produce a stronger, leak-free joint. In fact, very often the reverse is true. Overtightening can cause a leak in the joint and make it very difficult to reseal that particular joint when changing columns.

OPERATING SEQUENCE

Connecting a Glass Packed Column to a TCD and to a S/SL injector

Materials required:

- liner cap removal tool
- retaining nut
- ferrules
- metal liner

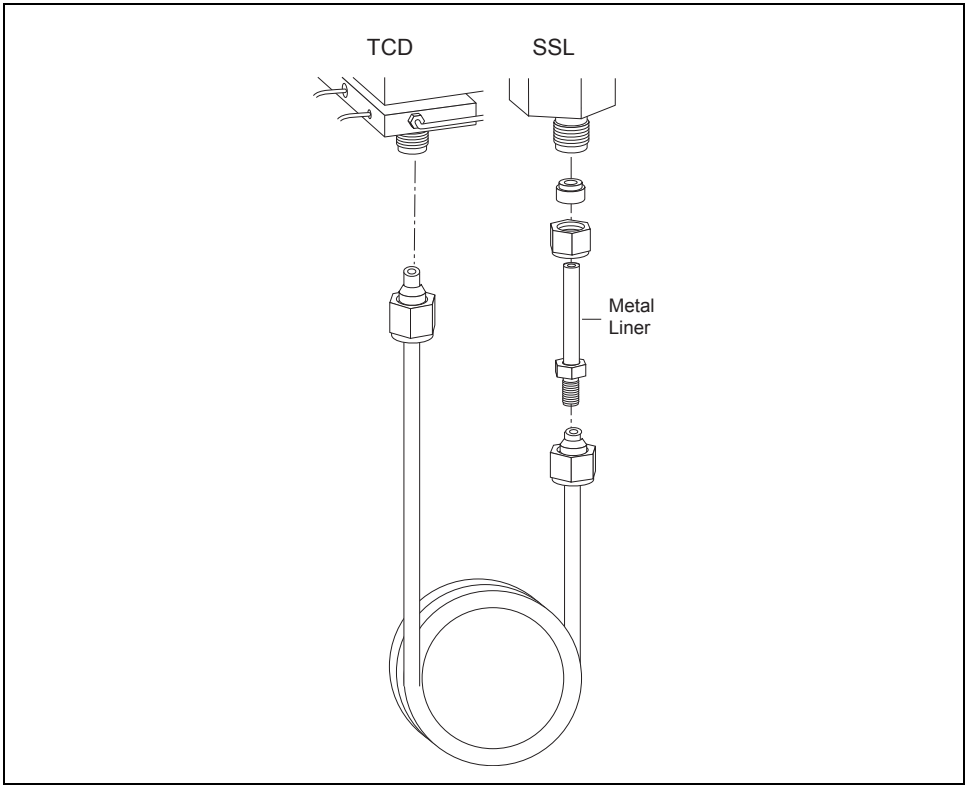
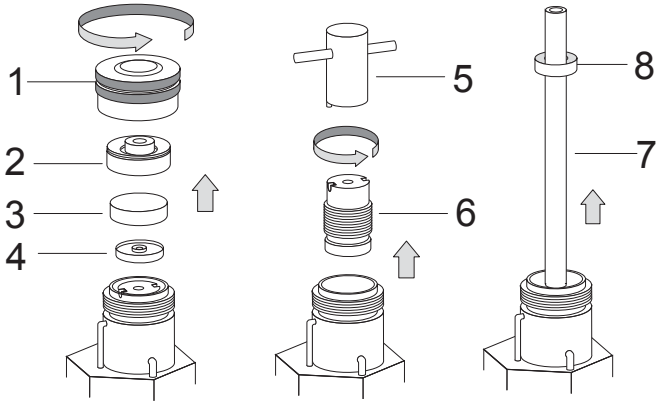


Figure 14-19. TCD-SSL Configuration

In this configuration the longer end of the column connects to the detector base and the shorter end connects to the injector as shown in Figure 14-19. The use of a metal liner is required.

Removing the S/SL Injector Top Components

With reference to Figure 14-20 proceed as follows:



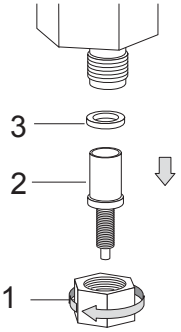
1. Injector Cap	2. Septum Holder
3. Septum	4. Septum Support
5. Liner Cap Removing Tool	6. Liner Cap
7. Liner	8. Graphite Seal

Figure 14-20. Removing the S/SL Injector Top Components

1. Unscrew the injector cap.
2. Remove the septum holder with septum. then the septum support.
3. Remove the liner cap by using the tool provided.
4. Use tweezers to remove the liner with the graphite seal.

Removing the S/SL Injector Bottom Components

With reference to Figure 14-21 proceed as follows:



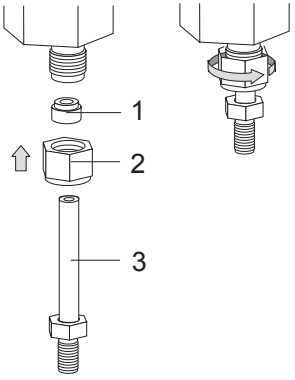
1. Terminal Fitting Retaining Nut
2. Terminal Fitting
3. Silver Seal

Figure 14-21. Removing the S/SL Injector Bottom Components

5. Unscrew the retaining nut at the bottom of the injector.
6. Remove the terminal fitting and the silver seal.

Installing the Metal Liner

With reference to Figure 14-22 proceed as follows:



1. Ferrule
2. Retaining nut
3. Metal Liner

Figure 14-22. Metal Liner Installation

7. Slide the appropriate nut and ferrule onto the metal liner, then insert it into the bottom of the injector.

8. Push the metal liner into the injector as far as possible.

9. Slide the ferrule up the injector base then finger-tighten the retaining nut until it starts to grip the metal liner.

10. Slide the appropriate graphite seal and push it onto the metal liner from the top of the injector by using the appropriate tool as shown in Figure 14-23.

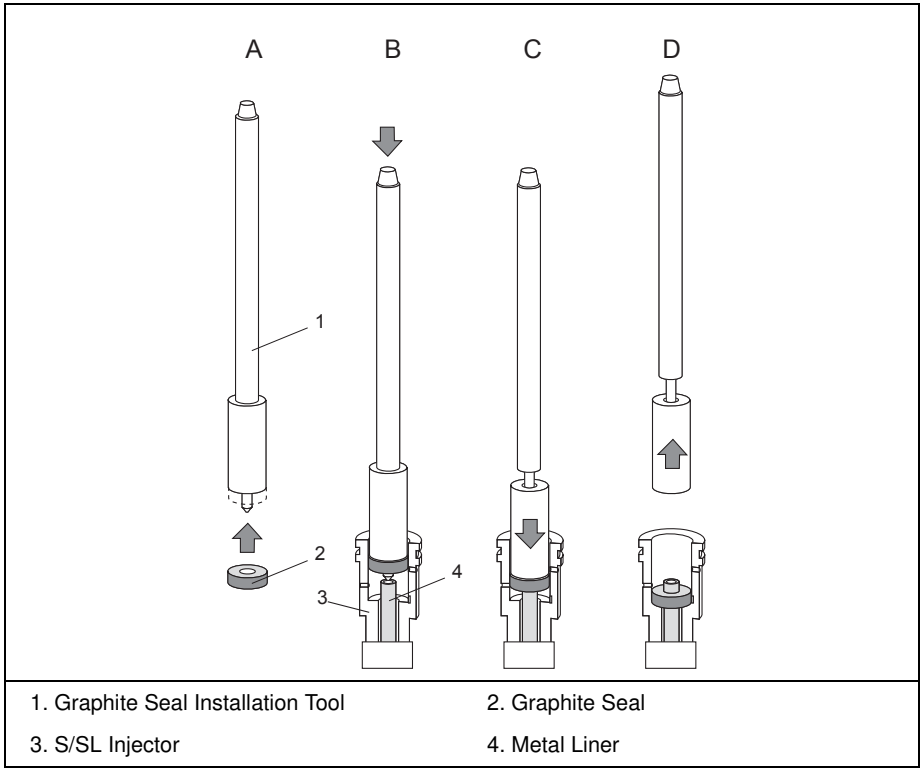


Figure 14-23. Graphite Seal Installation Tool

Connecting the Glass Packed Column

With reference to Figure 14-24 proceed as follows:

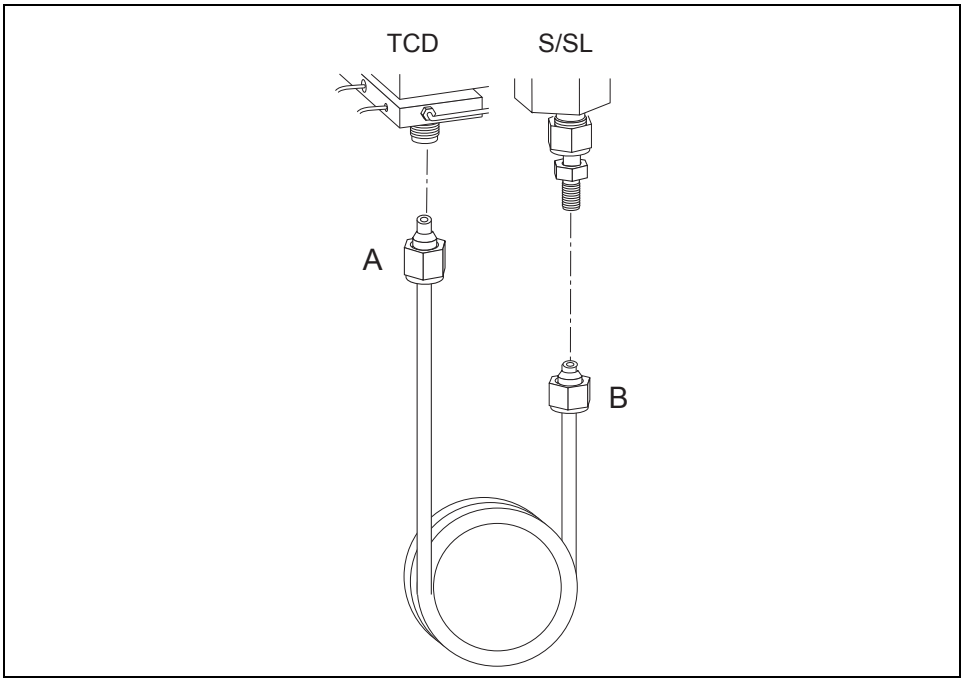


Figure 14-24. Connecting the Glass Packed Column

11. Make sure that your packed column has been correctly prepared as described in the *Preparing a Glass Packed Column* operating procedure on page 317.

12. Insert the column end A into the detector body and connect the column end B to the metal liner paying attention that the column end A touches the bottom

13. Finger-tighten the column ends A and B retaining nuts until they start to grip the column.

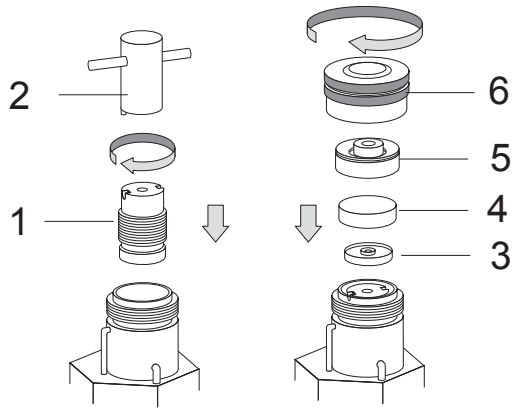
14. Finger-tighten the metal liner retaining nut.

CAUTION Overtightening the compression ferrule does not necessarily produce a stronger, leak-free joint. In fact, very often the reverse is true. Overtightening can cause a leak

in the joint and make it very difficult to reseal that particular joint when changing columns.

Reinstalling the S/SL Top Components

With reference to Figure 14-25 proceed as follows:



1. Liner Cap	2. Liner Cap Removing Tool
3. Septum Support	4. Septum
5. Septum Holder	6. Injector Cap

Figure 14-25. Reinstalling the S/SL Injector Top Components

15. Reinstall and tighten the liner cap until it start to grip the graphite seal.
16. Reinstall the septum support, septum, septum holder then screw the injector cap.

Keeping Column Flow Under Control

This paragraph describes the operations needed to perform the assisted Leak Check procedure and the Column Evaluation.

DCC-equipped System

TRACE GC Ultra, equipped with a DCC (Digital Carrier Control) module features a new user interface menu which guides the user through a sequence of operations necessary to correctly keep under control the pneumatic conditions of the GC system. The sequence *Leak Check - Column Evaluation* is the key for minimizing troubles related to leaks and correctly characterize the column pneumatic resistance.

- Perform **Leak Check** to assure the tightness of the system.
- Perform **Column Evaluation** to enter column information and, if a high flow accuracy is desired, to make a correction according to the actual flow of the carrier gas measured at the outlet of the column.
  During the column evaluation procedure, the system uses the correlation between the applied pressure, the flow, the column temperature. This operation allows to determinate the actual column pneumatic resistance and automatically correct the nominal column dimension (specifically the column ID). Column information must be entered every time a new column is installed.

Leak Check and Column Evaluation can be easily performed by means of the provided column-flow meter connector shown in Figure 14-26.

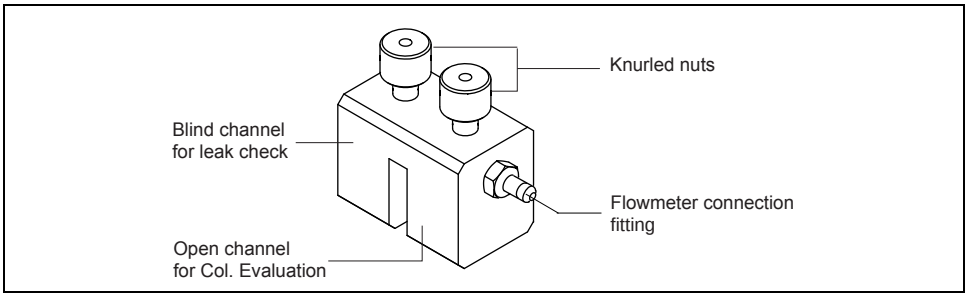


Figure 14-26. Column-flow Meter Connector

The column-flow meter connector features two dedicated channels:

- A **blind channel** used to seal the column end when a **leak check** is performed.
- An **open channel** used to measure the flow at the end of the column when a **column evaluation** is performed. A proper fitting permits an easy connection of the flowmeter.

Leak Check

Perform the *Leak check* at the desired pressure following the instructions reported in *Performing a Leak Check* operating sequence.

OPERATING SEQUENCE

Performing a Leak Check

Before starting this sequence, install the column into the injector port only, leaving free the column end.

Materials needed:

- Column-flow meter connector

1. Confirm that the carrier gas is on.
2. Carefully push the capillary column end into the **blind channel** of the column-flow meter connector as shown in Figure 14-27.

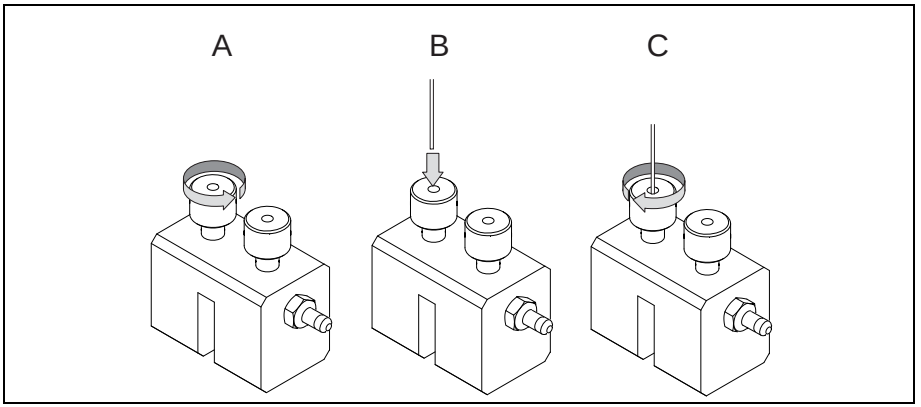


Figure 14-27. Leak Check

3. Press LEAK CHECK to enter **Leak Check** menu. The displayed options (`Right column` or `Left column`) depend on the configuration of your GC. In the example, the left column is considered.

```
     LEAK CHECK COLUMN
Right column[1]
Left column[1]                <
```

1. This item appears if the relevant channel is present and configured.

4. Scroll to the channel of interest and press ENTER to open the following menu:

```
     L. COL. LEAK CHECK
Start Leak Check
Leak Check Settings
```

5. Scroll to **Leak Check Settings** and press ENTER to open the relevant menu.

```
       L. COL. SETT.
Check press           200
Sensitivity           5.0
```

6. In **Check press** line set the pressure (pressurization value) at which the leak check must be performed. The range is comprised from 10 to 999 kPa (1.45-145 psi).

7. In **Sensitivity** line set the maximum value the pressure can drop during the test. The range is comprised from 1 to 10 kPa (0.145-1.45 psi).

8. Press CLEAR to exit **Leak Check Settings** menu and return to the previous menu.

```
     L. COL. LEAK CHECK
Start leak check
Leak Check settings
```

9. Select the **`Start leak check`** command to start the operation. The split and purge valves of the selected channel are automatically closed and the channel is pressurized with carrier gas to the leak check set point.

```
     CHECKING L COLUMN
Pressure              (200)
Elapsed time          0.90
Use <STOP> to abort
```

To abort Leak Check, press STOP. A relevant message will be displayed.

10. The system is pressurized for one minute, then the pressure value of the carrier is automatically set to **Off.** The system monitories the pressure for one minute. During this time, if the pressure **does not drop** more than the **Sensitivity** set value, the message **Leak check passed** is displayed.

```
      R/L.LEAK CHECK
        COMPLETED
       SUCCESSFULLY
    Leak check passed.
```

If not, the message **Leak detected** is displayed indicating possible leaks in the system. Locate and eliminate the leaks and repeat the leak check procedure.

Column Evaluation

Perform the *Column Evaluation* following the instructions reported in *Performing a Column Evaluation* operating sequence.

OPERATING SEQUENCE

Performing a Column Evaluation

Materials needed:

- Column-flow meter connector
- Thermo Scientific GFM Pro Flowmeter or equivalent, or soap bubble flowmeter

NOTE

A digital flowmeter capable to measure low flows down to 0.5 mL/min with an accuracy of ± 0.2% is recommended for an accurate column evaluation. The Thermo Scientific GFM Pro Flowmeter is recommended for the required accuracy of the flow measurement.

1. Confirm that the carrier gas is on.
2. On the GC keypad, press COLUMN EVAL to open the following menu:

```
     COLUMN INFORMATION
Right column
Left column              <
```

3. Scroll to the `Right` or `Left column` to evaluate and press ENTER. The following menu appears. In the example `Left column` is considered.

```
     LEFT[1] COLUMN INFO
Length (m)           15.00
ID (mm)               0.25
Film th. (µm)         0.25
```

1. These settings could also be for a right column.

4. Set the nominal dimensions of the column:
   - Length in the range from 0.01 to 200 m
   - Internal diameter in the range from 0.050 to 0.999 mm
   - Film thickness in the range from 0.01 to 20 µm
5. Select `Pre/post column?` yes (`Y`) or not (`N`).
   - If a pre-/post-column is not present, select **`N`**.
   - If a pre-/post-column is present, select **`Y`**. The menu requires to set the length and the nominal internal diameter of the pre-/post-column in the same ranges valid for the column. The following two lines are added to the menu.

```
Pre/post column?          Y
P/p col. L            10.00
P/p col. ID            0.53
```

6. According to the nominal dimensions of the column, the system calculates and shows the theoretical Column K-factor.

```
     LEFT[1] COLUMN INFO
Column  K           (0.8087)
Calc'd ID           (0.0000)
Run Column eval?
```

1. These settings could also be for the right column.

Theoretical column k-factor can be used to operate in flow mode. However, the accuracy of the calculated carrier flow rate will depend on the accuracy of the nominal column dimensions versus the actual values. In particular, the deviation of the actual column ID from the nominal value is mostly affecting the column flow rate. To assure the utmost accuracy for the column carrier flow rate calculation, the Column Evaluation procedure is recommended.

7. Carefully push the capillary column end into the **open channel** of the column-flow meter connector as shown in Figure 14-27.

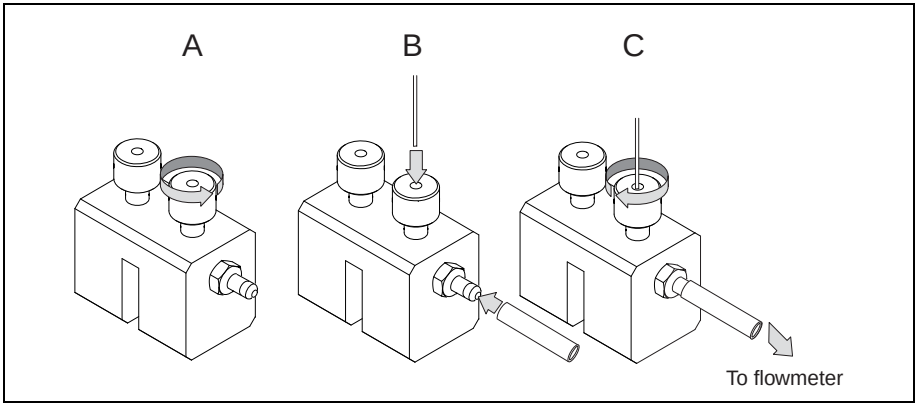


Figure 14-28. Flow Measurement

8. Connect the flowmeter to the dedicated fitting on the column-flow meter connector.

9. To start column evaluation, scroll to `Run column eval.?` and press ENTER.

WARNING! When a MS detector is used and the Vacuum Compensation parameter is set ON in Carrier Menu, the column evaluation cannot be performed. A message will alert for switching the Vacuum Compensation to Off.

The system, according to the theoretical k-factor (`Column K`), pressurizes the column to obtain a carrier flow of 5 mL/min. The maximum pressure set value is 500 kPa even if the required pressure would be higher. The minimum pressure set value is 10 kPa even if the required pressure would be lower. The display visualizes the pressure setting during the evaluation procedure.

10. By using the flowmeter, measure the carrier gas flow rate at the outlet of the column. Scroll to `Measured Flow` and enter the measured value.

```
    EVALUATING L COLUMN
Pressure              (162)
Measured Flow:        4.90
Use <STOP> to abort
```

NOTE To abort Column Evaluation, press STOP. A relevant message will be displayed.

11. The following message will be displayed in case of successful operation.

```
    L. COL. EVALUATION
        COMPLETED
      Calc'd ID 0.242
         K. 0.9020
```

The theoretical k-factor is automatically corrected with the more accurate experimental k value. This corrected k-factor is used to correct the nominal column ID. An averaged calculated ID is displayed, which reflects the actual column pneumatic resistance, permitting a more accurate calculation of the carrier gas linear velocity.

Column Conditioning

CAUTION When conditioning a column, remove the column from the detector base body. If this is not possible, such as when using packed columns, you must remove the detector and jet, if present, from the detector base body.

Column conditioning consists of passing a carrier gas flow through the column and heating the column to a temperature 20–50 °C above the maximum temperature that will be used for running analyses, provided that temperature is within the operating range of the column.

For detailed information on column conditioning of your specific column, refer to the column manufacturer's instructions.

SECTION

V

Detectors

This section contains information about detector configuration and operation.

Chapter 15, *Detector Overview*, gives basic information about the detectors available with the TRACE GC Ultra.

Chapter 16, *Flame Ionization Detector (FID)*, describes the operating principles and sequences for the Flame Ionization Detector (FID).

Chapter 17, *Electron Capture Detector (ECD)*, describes the operating principles and sequences for the Electron Capture Detector (ECD).

Chapter 18, *Nitrogen Phosphorus Detector (NPD)*, describes the operating principles and sequences for the Nitrogen Phosphorus Detector (NPD).

Chapter 19, *Photoionization Detector (PID)*, describes the operating sequences and principles for the Photoionization Detector (PID).

Chapter 20, *Flame Photometric Detector (FPD)*, describes the operating principles and sequences for the Flame Photometric Detector (FPD).

Chapter 21, *Thermal Conductivity Detector (TCD)*, describes the operating principles and sequences for the Thermal Conductivity Detector (TCD).

Chapter 22, *Pulsed Discharge Detector (PDD)*, describes the operating sequences and principles for the Pulsed Discharge Detector (PPD).

15

Detector Overview

This chapter gives basic information about the detectors available with the TRACE GC Ultra.

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Detector Base Body 341
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Make-up Gas 344
Detector and Make-up Gas Configuration 345
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Detector Signal Menu 351

Operating Sequences

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Configuring an Auxiliary Detector 349
Programming the Auxiliary Detector 350
How to Use Baseline Compensation 353

Detector Configuration

The following detectors are available for the TRACE GC Ultra:

- Flame Ionization Detector (FID)
- Electron Capture Detector (ECD)
- Nitrogen Phosphorus Detector (NPD)
- Photoionization Detector (PID)
- Flame Photometric Detector (FPD)
- Thermal Conductivity Detector (TCD)
- Pulsed Discharge Detector (PPD)

The TRACE GC can be configured for up to three detectors of different types. Each detector is installed on the proper left or right detector base body (**LEFT DETECTOR**, **RIGHT DETECTOR**) . The third, or auxiliary, detector can be installed and configured as *Auxiliary* (**AUX DETECTOR**) to allow the following possible configurations.

- Stacked Analytical Configuration
- Dual FPD (twin tube) Configuration
- Third Detector Base Body Configuration

For further details refer to paragraph *Auxiliary Detectors* on page 347.

Each detector is controlled by an electronic board inserted into the appropriate slot (A, B, or C) in the electronic compartment of the GC. The type of detector and the make-up gas are already configured. Each detector can be configured for a specific make-up gas depending on the analytical requirements.

Detector Base Body

The detector options are fully and easily interchangeable because of *base bodies* that act as a bridge between the detector and analytical column.

The detector base body is available in two configurations:

- detector base body for packed columns.
- detector base body for capillary columns.

Packed Column Detector Base Body

This detector base body, shown in Figure 15-1, accepts glass and metal packed columns with outside diameters of up to 6 mm or 1/4 inch. The column enters the compartment right up to the base of the detector jet which sits at the top of the base body. Hydrogen and make-up gas flow past the end of the column. This minimizes dead volumes and column band broadening.

Capillary Column Detector Base Body

This detector base body, shown in Figure 15-2, can accept all types of capillary columns. The column enters the detector jet directly to eliminate any dead volumes. The base body allows columns to be connected using either M4 or M8 1 mm fittings.

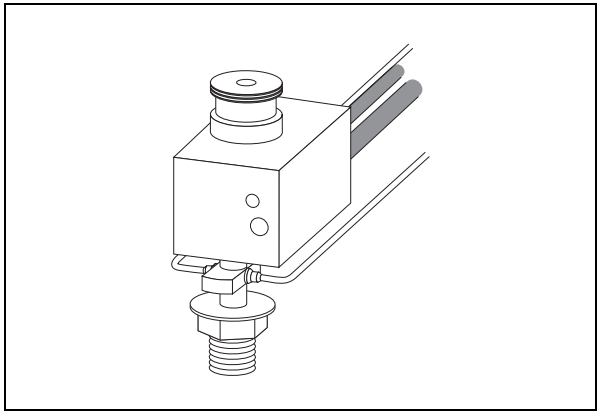

Figure 15-1. Packed Column Base Body

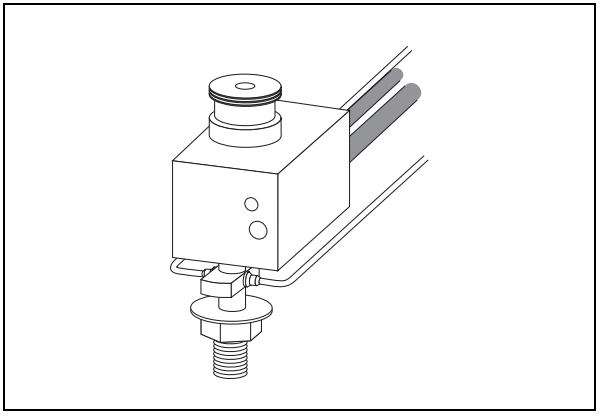

Figure 15-2. Capillary Column Base Body

Detector Gases

The GC automatically recognizes the detectors and detector gas modules installed. Different gas flow modules can be used for different detectors, but some detectors, such as the TCD, require specific modules. For more information about the different DGFC modules, refer to *Plumbing Detector Gases* on page 342.

The detector pneumatic control module supports up to four different modules, as shown in Table 15-1.

Table 15-1. Detector Module Gas Paths

	Detector Gas Path		
Controlled Module	Hydrogen	Air	Make-up
Type AA	—	—	X
Type AB	X	X	—
Type AC	X	X	X
Type AD	X	X	X

Plumbing Detector Gases

Different detector modules have different gas plumbing requirements. It is important that you connect the right gases to the right inlet fittings. The inlet fittings on the detector modules are labeled. To ensure that you have the detector gases properly connected, do the following:

1. Press INFO/DIAG twice to enter the **DIAGNOSTICS** menu.
2. Scroll to `Hardware config` and press ENTER.
3. In the **HARDWARE CONFIG** submenu, scroll to `L Det module`, `R Det module`, and `Aux Det module` (if configured) and note the module type.

4. Consult Table 15-2 for the proper gas connections of the detector modules installed.

WARNING! Hydrogen is a potentially dangerous gas. Always perform a leak check of the hydrogen gas line. Refer to *Using Hydrogen* on page xxviii for safety information.

Table 15-2. Detector Gas Connections

Detector	Installed Module	Connect Hydrogen to	Connect Air to	Connect Make-up Gas to[1]	Connect Sheath Gas to[2]	Connect Reference Gas to	Helium from purifier
FID	AB	Gas 2	Gas 1	—	—	—	
	AC	Gas 2	Gas 1	Gas 3	—	—	
	AD	Gas 3	Gas 1	—	—	—	
ECD	AA	—	—	Gas 3	—	—	
	AB	—	—	Gas 2	—	—	
	AC	—	—	Gas 3	—	—	
	AD	—	—	Gas 3	—	—	
NPD	AD	Gas 2	Gas 1	Gas 3	—	—	
PID	AB	—	—	Gas 2	Gas 1	—	
	AC	—	—	Gas 3	Gas 1	—	
	AD	—	—	Gas 3	Gas 1	—	
FPD	AB	Gas 2	Gas 1	—	—	—	
	AC	Gas 2	Gas 1	Gas 3[3]	—	—	
	AD	Gas 3	Gas 1	—	—	—	
TCD	AB	—	—	Gas 2	—	Gas 1	
PDD	dedicated						Inlet

1. For ECD detectors, the makeup gas is N_2 or 5% Ar/CH_4.
2. For PID detectors, the sheath gas is N_2 or He.
3. FPD applications typically do not require Make-up gas.

Make-up Gas

Most detectors require an auxiliary gas flow to improve sensitivity and peak shapes. This *make-up* gas helps to rapidly sweep the compounds from the column through the detector. The make-up gas you use depends on the detector. The `Make-up gas` parameter of the detector menu changes depending on your GC's configuration.

Detector and Make-up Gas Configuration

You configure the detectors and make-up gases in the **CONFIGURE** menu. The **LEFT** and **RIGHT DETECTOR** menus change to reflect the choices you make in the **CONFIGURE** menu.

Press CONFIG, then scroll to `Left Detector` or `Right detector` and press ENTER to open the detector gas menu.

Table 15-3. Configure Detector and Make-up Gas Menu

Menu	Submenu	Comments
`LEFT DETECTOR`		This line is the menu title bar.

`Detector type`	`DETECTOR TYPE`
	`* XXX-A <`
	`---------`
	`None`
		This indicates the type of detector mounted and the slot position (A, B, or C) of the relevant electronic control board. Select `Detector type` and press ENTER to display the submenu. An asterisk appears beside the detector selected.

`Makeup gas`	`MAKEUP GAS (XX)`
	`* Helium <`
	`Nitrogen`
	`Hydrogen`
	`Argon Methane 5%`
	`Argon`
	`None`
		This line appears only if the DGFC module is present. The type of make-up gas currently used for the detector is shown. Different suitable make-up gases may be selected depending on the type of detector installed. Table 15-4 shows the commonly-used make-up gases.
		Select `Makeup` and press ENTER to open the **MAKEUP GAS** submenu. Only the gases applicable to the detector in use are displayed.
		In the submenu, an asterisk appears beside the currently-active make-up gas. The active make-up gas is also displayed in parentheses in the menu title bar.

Table 15-4. Make-up Gases

		Detector					
		FID	ECD	NPD	PID	FPD	TCD
Gas	Helium	X		X	X	X	X
	Nitrogen	X	X	X	X	X	X
	Hydrogen	---	---	---	---	---	X
	Argon/5% Methane	---	X	---	---	---	
	Argon	---	---	---	---	---	X

OPERATING SEQUENCE

Configuring the Detector and Make-Up Gas

1. Press CONFIG and scroll to `Left Detector` or `Right Detector`, depending on the location of the detector you want to configure.
2. Select `Detector type` and press ENTER.
3. To change the detector type, scroll to the desired detector and press ENTER to confirm the selection. An asterisk appears beside the detector selected.

   To deactivate the detector, scroll to `None` and press ENTER.
4. Scroll to `Makeup` and press ENTER. The gases applicable to the detector in use are displayed.

   An asterisk appears beside the currently active make-up gas. The active make-up gas is also displayed in parentheses in the menu title bar.
5. To change the make-up gas, scroll to the desired gas and press ENTER. An asterisk appears beside the make-up gas selected.

   To deactivate the make-up gas, scroll to `None` and press ENTER.

Auxiliary Detectors

A detector is considered as *auxiliary* when it is not installed on the standard left or right detector base body position. The possible auxiliary detector configurations are the following.

Tandem (Stacked) Configuration

If you are using an ECD, which is a non-destructive detector, you can stack an *auxiliary* detector on top of it to operate in series. To stack an FID, NPD or FPD on top of the ECD, you must install a specially heated series adapter, as shown in Figure 15-3.

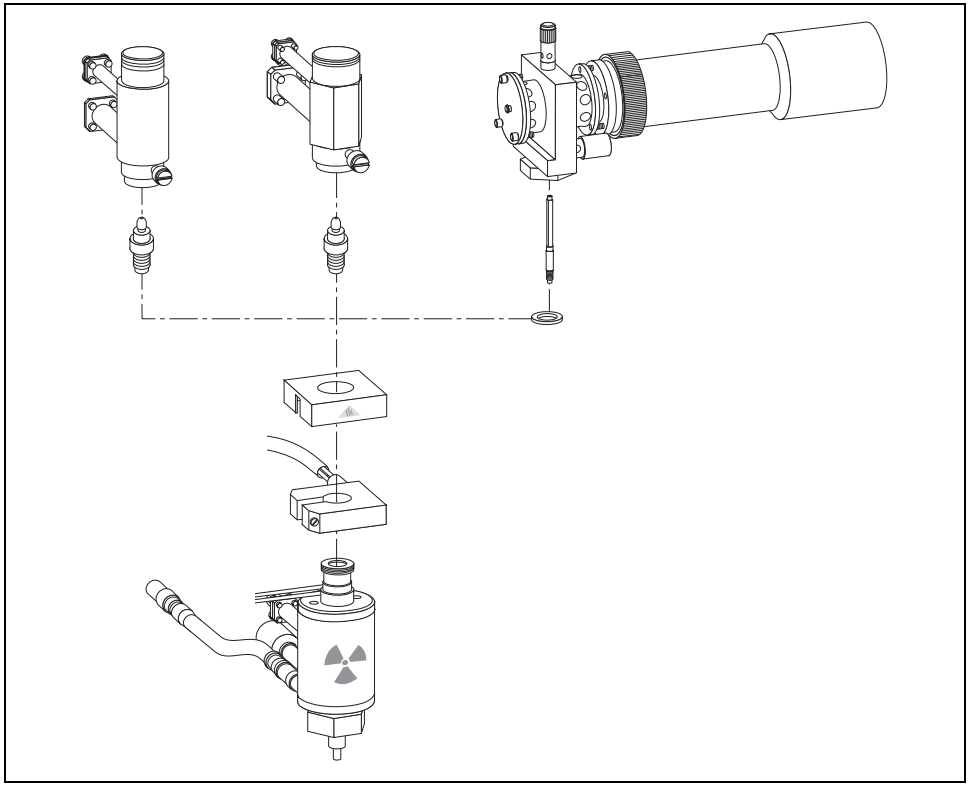

Figure 15-3. FID, NPD and FPD Series Connections to an ECD

The fuel gas for the auxiliary detector is supplied from an additional pneumatic module fitted in the pneumatic compartment. Your TRACE GC Ultra must be pre-configured at the factory if you plan to use an auxiliary detector.

Dual FPD Configuration

If you are using an FPD, you can expand it by connecting a second photomultiplier tube with different interferential filter on the same detector body. This allows to process a sample for phosphorous and sulphur (or tin) profiles simultaneously.

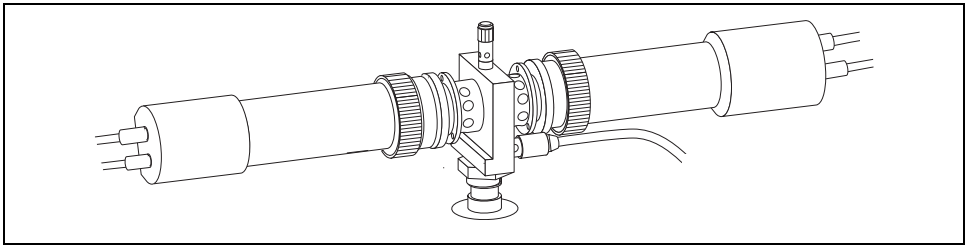

Figure 15-4. Dual FPD Configuration (Twin Tube)

In this configuration the FPD detector, already installed on the proper detector base body, is configured as **LEFT** or **RIGHT DETECTOR** while the second photomultiplier tube must be configured as **AUX DETECTOR**.

Note that the temperature and detector gases setpoints are common for both the photomultiplier tubes.

Third Detector Base Body

This configuration allows to install the third detector over an additional base body installed instead of an injector.
This configuration is permitted only for FID, NPD or PID.

OPERATING SEQUENCE

Configuring an Auxiliary Detector

Use the following sequence to configure an auxiliary detector and make-up gas.

1. Press CONFIG and scroll to `Auxiliary detector`.

NOTE The `Auxiliary detector` item will only be present in the **`CONFIGURE`** menu if your TRACE GC Ultra has been pre-configured at the factory for an auxiliary detector.

2. Scroll to `Detector type` and press ENTER.
3. In the **`DETECTOR TYPE`** submenu, scroll to the type of detector you want to use and press ENTER. Select `None` to deactivate the auxiliary detector.
4. If required, scroll to `Makeup gas` and press ENTER.
5. In the **`MAKEUP GAS`** submenu, scroll to the make-up gas you want to use with the auxiliary detector and press ENTER. Select `None` to deactivate the auxiliary detector makeup gas.

OPERATING SEQUENCE

Programming the Auxiliary Detector

Use the following sequence to set the parameters in the **AUXILIARY** menu.

1. Press AUX.
2. Scroll to Detector and press ENTER to display the **AUX DETECTOR** menu.
3. Configure the detector parameters in the menu. The **AUX DETECTOR** menu contains the same parameters as the **LEFT** and **RIGHT DETECTOR** menus. The parameters will change depending on the type of auxiliary detector you are configuring.

The parameters for the FID are described in *FID Menu* in Chapter 16.

The parameters for the NPD are described in *NPD Menu* in Chapter 18.

The parameters for the FPD and Dual FPD are described in *FPD Menu* in Chapter 20.

Detector Signal Menu

The **DETECTOR SIGNAL** menu contains the parameters that control the detector signal. As compounds elute from the column and enter the detector, an electrical signal is generated. The size of the signal is related to the amount of the corresponding compounds. The detector's electronics process the signal and send it to a recording device. The plot of the signal size versus the time results in the chromatogram.

Press LEFT SIGNAL or RIGHT SIGNAL to display the **SIGNAL** menu shown in Table 15-5. The **AUX SIGNAL** menu is identical to the **LEFT** and **RIGHT SIGNAL** menus.

Table 15-5. Detector Signal Menu

Menu	Range	Comments
LEFT SIGNAL (XXX)		This line is the title bar. The detector type is indicated in parentheses (XXX).
Output	Not editable	This is the actual electrometer output signal expressed in µV. The Autozero function forces this value to 1000 corresponding to the zero level of the baseline on a recording device. You cannot enter a setpoint here.
Offset	Variable, depending on detector output	This is a value in counts that may be subtracted from the Output signal to adjust the baseline level. This parameter may be manually or automatically set using the Auto zero function. The range of the suppression is variable and related to the output signal.
Auto zero?	Yes/No	This function forces the output signal to 1000 (zeroing). Press YES to zero the detector signal. The Auto zero in progress message is displayed.

Table 15-5. Detector Signal Menu (Continued)

Menu	Range	Comments
`Range 10^ (0...3)`[1]	10^0–10^3 (1, 10, 100, 1000 nA) for FID, NPD, PID and PDD 10^0–10^2 (1, 10, 100 nA) for FPD	This parameter sets the electrometer amplifier input range. 10^0 is the most sensitive.
`Gain (x1 or x10)`[2]	x1, x10	This parameter allows you to increase the amplifier gain by a factor of 10.
`Neg. polarity`[2]	Yes/No	This parameter allows you to reverse the polarity of the signal as a function of the thermal conductivity of the carrier gas.
`Analog filter`[2]	On, Off	This parameter allows output signal filtering to minimize the noise of the baseline. Press ON to enable the filtering. This also increases the response time of the detector.
`Baseline comp`	On, Off	This parameter allows the baseline compensation. This function is used when the subtraction of the baseline from the output signal is required; e.g. to subtract a blank analysis from the current one. When ON, it is enabled. When OFF it is no enabled. Depressing MODE/TYPE, the menu will be opened for setup. Refer to *How to Use Baseline Compensation* operating sequence.

1. This line is not displayed for the ECD or TCD.
2. This line is displayed only for the ECD, FPD and TCD.

NOTE

With FID and PDD if the `Range 10^` is set 2 or 3, the small variation of the output signal is not detected. For this reason, the, `Signal pA`, `Ign. thresh` and `Flameout retry` parameters will be not displayed in the **`DETECTOR FID`** menu and the `Signal pA`, parameter will be not displayed in the **`DETECTOR NPD, PID, PDD`** and **`FPD`** menus.

OPERATING SEQUENCE

How to Use Baseline Compensation

Use the following sequence to use baseline compensation parameter.

CAUTION When UFM module is used, the electrometric control card must be in the expansion slot marked A located on the left part of the GC mother board in the electronic compartment.

1. With the GC in stand-by/Ready to Inject condition, enter **`SIGNAL`** menu and perform the Autozero.

2. Scroll to `Baseline comp` and keep it OFF.
   Press MODE/TYPE to enter Baseline Compensation menu.

```
        BASELINE COMP

Setup comp run              <
Start comp run
Setup comp output
```

3. Select `Setup comp run` to define which detector baseline must be storage.
   Press ENTER, the following submenu is displayed:

```
        BASELINE COMP

Run R det comp             On<
Run L det comp             On
Run Aux comp[1]            Off
```

1. This line is displayed only when Auxiliary Detector is configured.

4. Turn on the detector of which the baseline compensation is required. Up to three detectors compensation can be simultaneously carried out. Press CLEAR to exit the submenu.

5. Select `Start comp run`. Press ENTER, to begin the collection of the data to use for the baseline compensation. The following message is displayed:

```
      Baseline comp run
        in progress

       Collecting Data
```

6. At the end of the data collection the following message should be displayed; if not repeat the procedure.

```
      Baseline comp run
          complete

Data OK
```

Press CLEAR up to return to **`SIGNAL`** menu. Scroll to `Offset` and turn it OFF.

7. Scroll to `Baseline comp`. Press MODE/TYPE to enter Baseline Compensation menu. Select `Setup comp output` to define the detector output from which the baseline must be subtracted. Press ENTER, the following submenu is displayed.

```
      SUBTRACTED OUTPUT

Right detector          On <
Left detector           On
Aux detector[1]         Off
```

1. This line is displayed only when Auxiliary Detector is configured.

8. Turn on the detector from which the baseline must be subtracted from the output. Up to three detectors can be set.

NOTE

The start for collecting data or baseline subtraction must be programmed also through the sequence programming, or through the Clock Table Programming.
For details, refer to *The Clock Table* in Chapter 24.

16

Flame Ionization Detector (FID)

This chapter describes the Flame Ionization Detector (FID). Due to its high sensitivity, good operational stability, and wide linear response, the FID remains the most popular detector for gas chromatography.

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FID Overview 355
FID Gas Supplies 357
FID Installation 359
FID Menu 361

Operating Procedures

Programming an FID 363
Setting the FID Signal Parameters 364

FID Overview

In the FID, 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 the metal collar surrounding the flame forms the collecting electrode.

A polarizing voltage is applied across the electrodes from the electrometer unit to accelerate and collect the ions that are generated during the combustion process of

organic compounds. The resulting ionization current is sensed by an electrometer amplifier and converted to a suitable output signal. Figure 16-1 shows the FID.

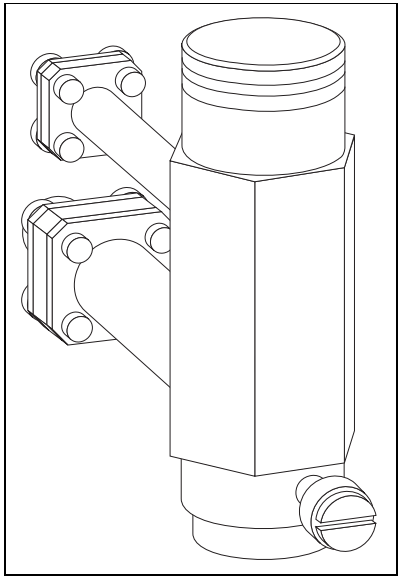


Figure 16-1. Flame Ionization Detector

Jet

The flame jet, mounted on the detector base body for capillary, wide-bore, or packed columns, is suitable for operating temperatures of up to 450 °C. It has ceramic insulation.

Selectivity

The FID responds to almost all organic compounds containing a carbon-hydrogen bond. The detector does not respond, or responds minimally, to a number of compounds such as permanent gases, oxides of nitrogen, sulfur compounds, ammonia, and water.

Temperature

The detector base body heats the FID. Its exact temperature is not critical. It only has to be sufficiently high to prevent condensation of the water vapor formed as a

result of the hydrogen combustion of the flame. It cannot be used with a detector base body temperature of less than 150 °C. The TRACE GC Ultra will not allow flame ignition to proceed at temperatures less than 150 °C. The base body temperature is normally set to the upper temperature limit of the column in use.

FID Gas Supplies

The stability and analytical performance of the FID is greatly affected by the flow of the various gases through the detector.

The gases normally used with the FID are shown in Table 16-1.

Table 16-1. FID Carrier Gases.

Carrier Gas	Capillary Columns	Packed Column
Helium	X	X
Nitrogen	X	X
Hydrogen	X	
Argon		X

The carrier gas flow range depends on the type of gas used and on the type and diameter of the capillary or packed column installed.

The fuel and make-up gases used for the FID are:

- fuel gas:hydrogen and air
- make-up gas:nitrogen (recommended) or helium

Make-up gas is not required when a packed column is used.

The recommended ranges of detector gas flow rates tolerated by the FID are:

- hydrogen:30–50 mL/min
- air:300–600 mL/min
- make-up gas:10–60 mL/min

Usually the air flow is about ten times the hydrogen flow to keep the flame lit.

To gain optimal performance from the FID, you should experiment with the hydrogen flow rate, keeping the carrier and air flows constant, to obtain the maximum signal intensity for the components of interest.

For high sensitivity applications, it is essential to exclude all traces of organic contamination from the chromatographic system and/or detector gas lines. Such contamination may create ghost peaks in the chromatogram or, more often, an unstable baseline. Table 16-2 shows typical FID operating conditions.

Table 16-2. Typical FID Operating Conditions

Parameter	Capillary Columns	Packed Columns
Base temperature	250 °C	250 °C
Carrier	2 mL/min	40 mL/min
Hydrogen	35 mL/min	40 mL/min
Air	350 mL/min	500 mL/min
Make-up gas (Nitrogen)	30 mL/min	Not used

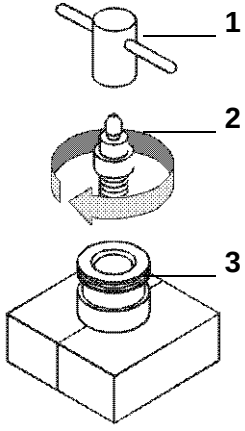
FID Installation

This operation allows the correct installation of the FID on your TRACE GC Ultra.

Material required

- Jet for FID
- Tool for jet

1. Place the jet into the detector base body housing and tighten it with the proper tool. Ensure the jet is perfectly vertically aligned to avoid damaging its ceramic part.

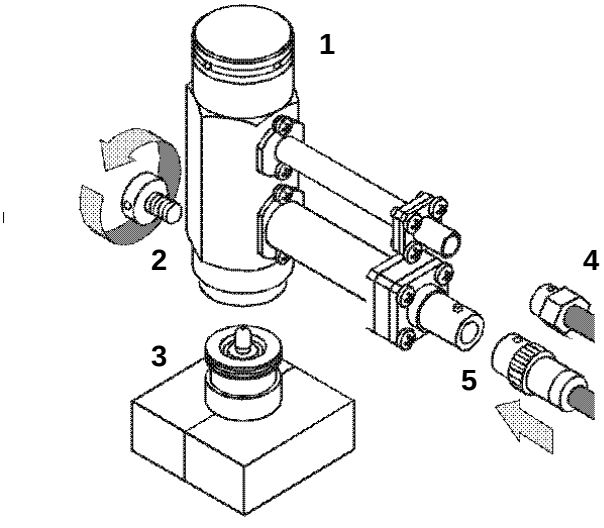


1. Tool for jet
2. Jet
3. Detector base body

Figure 16-2. Jet for FID

2. Install the FID on the detector base body and secure it by using the fixing screw on the front of the detector cell.

3. Carefully, connect the signal and ignition polarization cables coming from the detector control card, to the detector cell.



1. FID	2. Fixing screw
3. Detector base body	4. Signal cable
5. Ignition polarization cable	

Figure 16-3. Installation of the FID

FID Menu

The **DETECTOR (FID)** menu contains the detector control parameters if the GC has been configured for an FID. Press LEFT DETECTOR or RIGHT DETECTOR to open the menu shown in Table 16-3.

Table 16-3. Detector (FID) Menu

Menu	Range	Comments
RIGHT DET (FID)		This line is the title bar.
Flame	On/Off	This indicates the flame status: On, Off, Igniting, or Out. Hydrogen and air flows are required to light the flame. Press ON to turn on the hydrogen and air flows. This happens only if the Base temp is ≥ 150 °C. If not, an error message is displayed. The Igniting message is displayed during the flame ignition sequence. The Out message is displayed when the flame is inadvertently extinguished. The **Not Ready** LED will be lit and the hydrogen and air supplies will automatically turn off. Refer to *Flame Out Conditions* on page 362 for more information. Press OFF to turn off the hydrogen and air flows.
Base temp	On/Off, 50–450 °C	This indicates the detector base body temperature. Press ON to turn on the heater and display the actual and setpoint values. Press OFF to turn off the heater and display the actual value.
Signal pA	Not editable	This parameter shows the collector current in picoamperes (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.

Table 16-3. Detector (FID) Menu (Continued)

Menu	Range	Comments
`Ignition thresh`	0.0–9.9 pA	The FID produces a small signal current when lit. This parameter defines the flame on condition. The TRACE GC Ultra uses this value to determine flame status (on or off) and control automatic re-ignition. If `Flameout retry` is `On`, the flame will re-ignite if the signal drops below this value.
`Flameout retry`	On/Off	This indicates re-ignition status. Press ON to program when the flame re-ignition should be attempted. Refer to *Flame Out Conditions* for more information.

`H2`
 	
`Air`
	On/Off, 0–200 mL/min for H_2
	
	On/Off, 0–600 mL/min for Air
		These indicate the hydrogen and air flow supplied to the detector. Press ON to turn on the gas flows and to display the actual and setpoint values. Press OFF or 0 to turn off the flows and to display the actual value. These flows can be turned on independently when the flame is off, but they are cut off when the flame is turned off, or when the FID fails the ignition sequence.

`Mkup (XX)`	On/Off, 0–100 mL/min	This indicates the make-up gas used with the FID. The type of gas is displayed in parentheses. Press ON to turn on the make-up gas flow and to display the actual and setpoint values. Press OFF or 0 to turn off the flow. The flow turns off during the flame ignition sequence, then it turns back on before the ignition threshold test. The flow remains turned on when the flame is turned off.

Flame Out Conditions

When the flame is accidentally extinguished, either permanently because of exhausted fuel gas supplies or temporarily, the `Flame Out` message is displayed in the menu and a message is recorded in the **Run Log**.

If the `Retry` function is turned `On`, the system will attempt to re-ignite the flame up to three times.

OPERATING SEQUENCE

Programming an FID

Before you begin this operating sequence, do the following:

- Verify that all detector gases are connected, a column is correctly installed, and the system is free of leaks.
- Check the oven temperature and injector temperature.
- Check the carrier gas flow depending on the capillary or packed column in use.

WARNING! Hydrogen is a potentially dangerous gas. Refer to *Using Hydrogen* on page xxviii for safety information.

1. Press LEFT DETECT or RIGHT DETECT to open the **`DETECTOR (FID)`** menu.
2. Set the detector base body temperature. This must be greater than 150 °C to allow flame ignition.
3. Change the hydrogen flow rate, if desired, according to the analytical requirement.
4. Change the air flow rate, if desired, according to the analytical requirement.
5. Change the make-up gas flow rate, if desired. When a packed column is installed, the make-up gas is not used. Turn it `Off`.
6. When the detector base body is at the set temperature, scroll to `Flame` and press ON. This turns on the air and hydrogen flows and initiates the ignition sequence. The signal increases after the ignition. A sudden baseline deflection indicates that the flame is lit inside the detector. After a few seconds, the baseline should stabilize to the standing current level of the system.
7. Press LEFT SIGNAL or RIGHT SIGNAL to open the **`SIGNAL`** menu and verify the output signal.

Refer to the *Setting the FID Signal Parameters* operating sequence on page 364 for instructions on setting the signal parameters.

OPERATING SEQUENCE

Setting the FID Signal Parameters

1. Press LEFT SIGNAL or RIGHT SIGNAL to enter the detector **`SIGNAL (FID)`** menu.
2. Scroll to `Range 10^ (0...3)` and set the electrometer amplifier input range. `0` (10^0) is the most sensitive.
3. Turn `Analog filter` ON if you want to filter the output signal.
4. Scroll to `Autozero` and press ON.
5. If offset is required, scroll to `Offset` and enter a numeric value or press ON to recall the last offset from memory.
6. Turn `Baseline comp` ON if you want to compensate the baseline.

NOTE

If the `Range 10^` is set 2 or 3, the small variation of the output signal is not detected. For this reason, the, `Signal pA`, `Ign. thresh` and `Flameout retry` parameters will be not displayed in the **`DETECTOR FID`** menu.

17

Electron Capture Detector (ECD)

This chapter describes the operating principles and sequences for the Electron Capture Detector (ECD).

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Operating Sequences

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ECD Overview

The ECD has a low volume ionization chamber and increased contamination resistance which ensure high sensitivity and reliability. The detector consists mainly of a stainless steel cylinder housing a ^{63}Ni radioactive source.

The source acts as a cathode in the ionization cell while another cylindrical coaxial electrode acts as an anode (collecting electrode). Heat resistant material ensures effective insulation between the two electrodes and the detector body.

The detector is heated by a low voltage resistor controlled by an electronic thermoregulator. Figure 17-1 shows the ECD.

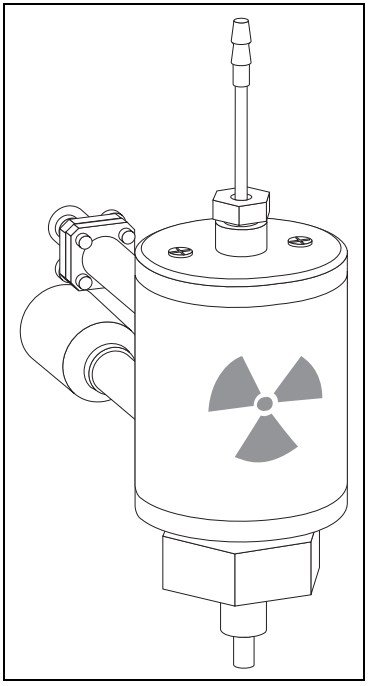


Figure 17-1. Electron Capture Detector

WARNING! The Electron Capture Detector (ECD) contains a ^{63}Ni beta-emitting radioactive source of 370 MBq (10 mCi).

The ^{63}Ni radioisotope, electrically deposited as metal on a nickel foil, is in a cylindrical source holder made of 6 mm stainless steel. This holder is fixed to the detector body, also made of stainless steel, to protect it and make it inaccessible from the outside.

The radioisotope is not released by its support at temperatures lower than 450 °C.

This temperature can never be reached by the detector, whose maximum operating temperature is 400 °C. A safety device (thermo-resistor regulator complying with standard DIN 43760) protects the detector and prevents overheating.

The normal operation of the detector does not involve any dispersion of solid or gaseous radioactive material, and therefore the risk of direct or secondary radiation (Bremsstrahlung) from the detector is practically nil.

The detector should never be opened or handled by the operator. Any maintenance or service operations involving even partial disassembling of the instrument must be performed ONLY by qualified personnel at a laboratory expressly authorized by Thermo Fisher Scientific and specifically licensed to handle radioactive material.

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 results of the values found.

The users of this detector in the United States are required to perform a wipe test on their ECD at intervals not to exceed 3 years (36 months) following the reported sequence. For other countries, please refer to the appropriate agency for their requirements.

ECD Gas Supplies

In the ECD cell, the ^{63}Ni source releases β particles that collide with the molecules of an easily ionizable carrier or make-up gas flowing through the detector 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 the ECD are shown in Table 17-1.

Table 17-1. ECD Carrier Gases

Carrier Gas	Capillary Columns	Packed Column
Helium	X	
Nitrogen	X	X
Hydrogen	X	
Argon/5% Methane		X

When using helium or hydrogen as a carrier gas with capillary or wide-bore columns, the detector should be fed with nitrogen or argon/methane through the make-up gas line.

WARNING! Hydrogen is a potentially dangerous gas. Refer to *Using Hydrogen* on page xxviii for safety information.

Operating Principle

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 [Equation (18-1)].

$$\beta^* + N_2 \Rightarrow \beta + N_2^+ + e^- \qquad (17\text{-}2)$$

A weak electrical field between the electrodes causes the electrons to collect rapidly at the anode and generate a small current (standing current). The possibility for *heavy* positive ions to recombine with electrons is negligible.

When an electron capturing substance passes through the detector cell, the current is reduced because of the absorption of electrons by this substance, according to one of the following reactions [Equations (18-2) and (18-3)].

$$e^- + AB \Rightarrow AB^- + h\nu \quad (17\text{-}3)$$

$$e^- + AB \Rightarrow A^{\cdot} + B^- \quad (17\text{-}4)$$

In Equation (17-2), an energized negative molecular ion forms, while in Equation (17-3), after the electron capture, the molecule dissociates (dissociative capture) generating a free radical $A^{\cdot}$ and a negative ion B^-.

The energy freed during the capture in Equation (18-2) is the measure of the electron affinity of the molecule.

The succession of phenomena determining the detector response ends with the neutralization of the negative ions formed by *capture*. The detector response is therefore related to the loss of electrons that occurs due to capture in the system.

The decrease in the electron concentration is converted into an electric signal proportional to the concentration of solute.

Molecular Structure and Detector Response

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, organometals, 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, as shown in Table 17-2. These values are also affected by temperature which enhances the detector response for those compounds capturing electrons dissociatively.

Considering the differences in response, you must calibrate the detector before performing quantitative determinations. To calibrate the detector, inject standard mixtures under the same operating conditions used for the samples to be tested.

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.

Table 17-2. Relative Response to Some Organic Compounds

Substance	Relative Sensitivity
Ethane Benzene	1
Butanol Acetone Chlorobutane Chlorobenzene	$1–10^2$
1,2 Dichloroethane Anthracene Keto-steroids Tetraethyl lead Benzyl chloride	$10^2–10^4$
Chloroform Nitrobenzene Carbon disulphide Cinnamaldehyde	$10^4–10^5$
Carbon tetrachloride Dinitrophenol Diethyl fumarate Dinotrobenzene Hexachlorobenzene Hexachlorocyclohexane	$10^5–10^6$

Constant Current Operating Mode

In the constant current, pulse-modulated mode, the detector is controlled by a PCB. During pulse application, electrons migrate to the anode, and therefore, their concentration in the cell rapidly drops to zero.

During the interval between pulses, electrons gradually return to their original concentration and to thermal equilibrium in which the capturing process is favorable.

In the relatively long interval between two short pulses, all electrons not consumed by capture are collected at the anode that measures the electron flow (cell current) present at that moment.

In Equation (18-4), the average cell current I is proportional to the concentration of electrons [e^-] collected at each pulse, and to the frequency of the applied pulses:

$$I = K[e^-]f \quad (17\text{-}4)$$

The cell current is forced to be constant, at a preset reference value, through an electric feed-back loop circuit that compares the cell current to the reference current at any time.

When an electron capturing compound enters the detector cell, the electron concentration [e^-] decreases and, according to Equation (17-4), the pulse frequency, required to collect the remaining free electrons, rises to maintain a constant cell current.

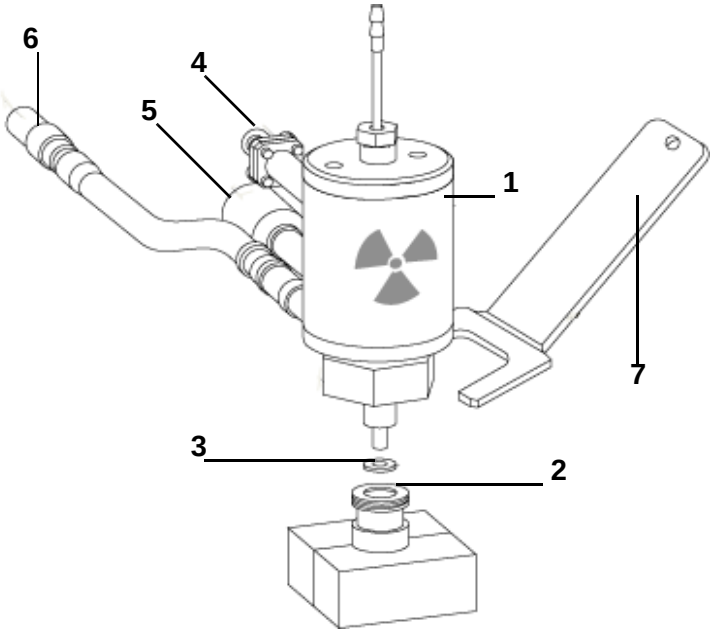
The difference in the frequency, when an electron capturing compound enters the cell, and the base frequency, when no sample is present, is converted into an electric signal which is proportional to the concentration of the compound in the detector.

ECD Installation

This operation allows the correct installation of the ECD on your TRACE GC Ultra. Refer to Figure 17-2.

Material required

- ECD Fixing Tool



1. ECD	2. Detector Base Body
3. Lower Seal	4. Signal connector
5. Excitation Connector	6. Temperature sensor/heater Connector
7. ECD Fixing Tool	

Figure 17-2. Installation of the ECD

1. Install the ECD on the detector base body interposing the lower seal. Secure the detector by using the ECD fixing tool

2. Carefully, connect the signal, excitation and temperature sensor/heater extension cables coming from the detector control card, to the detector cell.

ECD Menu

The **DETECTOR (ECD)** menu contains the detector control parameters if the GC has been configured for an ECD. Press LEFT DETECTOR or RIGHT DETECTOR to open the menu shown in Table 17-3.

Table 17-3. Detector (ECD) Menu

Menu	Range	Comments
RIGHT DET (ECD)		This line is the menu title bar.
Base temp	On/Off, 0–400 °C	This indicates the detector base body temperature. Press ON to turn on the heater and display the actual and setpoint values. Press OFF to turn off the heater and display the actual value.
ECD temp	On/Off, 0–400 °C	This indicates the detector temperature. Press ON to turn on the heater and display the actual and setpoint values. Press OFF to turn off the heater and display the actual value.
Ref current nA	0.0–3.0 nA in steps of 0.1 nA	This indicates the cell reference current expressed in nanoamperes.
Freq kHz	0–999.99 kHz	This indicates the actual value of the pulse frequency rate. Refer to *Base Frequency* on page 374 for more information.
Pulse amp V	5–50 V in a continuous mode	This indicates the pulse amplitude expressed in volts.
Pulse width us	0.1, 0.5, or 1.0 µs	This indicates the pulse width expressed in microseconds. Press ENTER to open the submenu. An asterisk appears beside the pulse width selected.
Mkup (XX)	On/Off, 0–100 mL/min	This indicates the make-up gas used with the ECD. The type of the gas is displayed in parentheses. Press ON to turn on the flow and display the actual and setpoint values. Press OFF the turn off the flow and display the actual value.

Base Frequency

Base frequency is an important parameter in evaluating the operating status of the ECD system.

For a constant concentration of thermal electrons inside the detector cell, the base frequency is a function of the reference current, pulse amplitude, and pulse width selected. The frequency increases when the reference current is increased or when the pulse duration or pulse amplitude is reduced.

For a given reference current, pulse duration, and amplitude, the base frequency remains constant when only carrier gas and make-up gas flow through the cell. The frequency generally increases, under the same operating conditions, because of decreased electron population inside the cell or reduced electron collecting efficiency. In the latter case, the collecting efficiency can be restored by cleaning or replacing the collecting electrode (anode).

If the electron concentration has decreased due to contaminants entering the detector cell, you must remove the source of contamination.With a high base frequency, the probability of electron capture tends to decrease, and therefore, the signal to noise ratio generally decreases.
You must select the appropriate reference current values to maintain the base frequency at acceptable levels in the **DETECTOR (ECD)** menu.

OPERATING SEQUENCE

Programming an ECD

Before you begin this sequence, do the following:

- Verify that all detector gases are connected, a column is correctly installed, and the system is free of leaks.
- Check the oven temperature and injector temperature.
- Check the carrier gas flow according to the capillary or packed column in use.

WARNING! Hydrogen is a potentially dangerous gas. Refer to *Using Hydrogen* on page xxviii for safety information.

1. Press LEFT DETECTOR or RIGHT DETECTOR to open the **`DETECTOR (ECD)`** menu.
2. Set the detector base body temperature.
3. Set the detector temperature. Keep in mind the maximum column temperature required for the analysis and the type of compounds to be detected. The ECD detector temperature is generally set between 250 °C and 350 °C.
4. Change the make-up gas flow rate, if desired.

NOTE During the heating stage, the make-up gas flow rate should be increased up to 50% over the normal operating flow rate.

5. Set the reference current to 1.0 nA.
6. Set a pulse amplitude of 50 V. If the GC system is ideally clean, a lower value can be selected to reduce the excitation level of electrons.
7. Scroll to `Pulse width` and press ENTER to open the submenu.
8. Select the desired pulse width depending on the gas in use and press ENTER.

   When nitrogen is used, a pulse width of 1.0 µs or 0.5 µs must be selected. 0.1 µs is recommended when using argon/methane.

9. Read the frequency value displayed. After you set a reference current of 1.0 nA, a pulse width of 1.0 µs, and a pulse amplitude of 50 V, a base frequency lower than 5 kHz should be displayed.

   Should the resulting frequency value be very low (1–2 kHz), the pulse voltage can be reduced to 15–30V and/or the pulse width can be set to 0.5 µs to increase the linear range and improve the signal to noise ratio.

10. Press LEFT SIGNAL or RIGHT SIGNAL to open the **`SIGNAL (ECD)`** menu. Verify the output signal.

Refer to the *Setting the ECD Signal Parameters* operating sequence on page 376 for instructions on setting the signal parameters.

OPERATING SEQUENCE

Setting the ECD Signal Parameters

1. Press LEFT SIGNAL or RIGHT SIGNAL to enter the detector **`SIGNAL (ECD)`** menu.

2. Scroll to `Auto zero?` and press ON.

3. If offset is required, scroll to `Offset` and enter a numeric value or press ON to recall the last offset from memory.

4. Turn `Baseline comp` ON if you want to compensate the baseline.

18

Nitrogen Phosphorus Detector (NPD)

This chapter describes the principles and sequences for the Nitrogen Phosphorus Detector (NPD).

Chapter at a Glance…

NPD Overview 377
Thermionic Source Lifetime 379
NPD Gas Supplies 379
NPD Installation 380
NPD Menu 382

Operating Procedures

Programming an NPD 384
Setting the NPD Signal Parameters 387

NPD Overview

The NPD provides selective detection of nitrogen or phosphorous-containing organic compounds. A ceramic matrix thermionic source, positioned above the jet, 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.

A thermionic source with a different surface coating is also available. This source provides high specificity and sensitivity to certain electronegative molecules when operating in an inert nitrogen gas environment. This is the Enhanced Nitrogen Selectivity (ENS) operating mode.

The jet, mounted on the detector base body, is suitable for operating temperature up to 450 °C. Figure 18-1 shows the NPD.

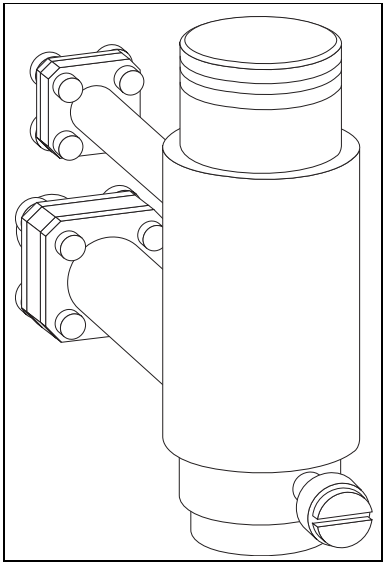


Figure 18-1. Nitrogen Phosphorus Detector

Thermionic Source Lifetime

Source lifetime 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 you turn 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.

NPD Gas Supplies

The gases normally used with the NPD are shown in Table 18-1.

Table 18-1. NPD Carrier Gases

Carrier Gas	Capillary Columns	Packed Column
Helium	X	X
Nitrogen	X	X
Hydrogen	X (only with DGFC)	

WARNING! Hydrogen is a potentially dangerous gas. Refer to *Using Hydrogen* on page xxviii for safety information.

The carrier gas flow range depends on the type of the gas used and on the type and diameter of the capillary or packed column installed.

The fuel and make-up gases for the NPD are:

- fuel gas: hydrogen, air
- make-up gas: nitrogen, helium

Nitrogen is preferred over helium because it has a much lower thermal conductivity and it requires a lower heating current for the source.

NOTE A make-up gas is not necessary when a packed column is used.

The detector gas flow rates generally used are:

- hydrogen: 2–4 mL/min
- air: 40–80 mL/min
- make-up: 10–20 mL/min

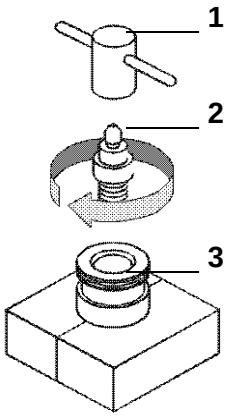
NPD Installation

This operation allows the correct installation of the NPD on your TRACE GC Ultra.

Material required

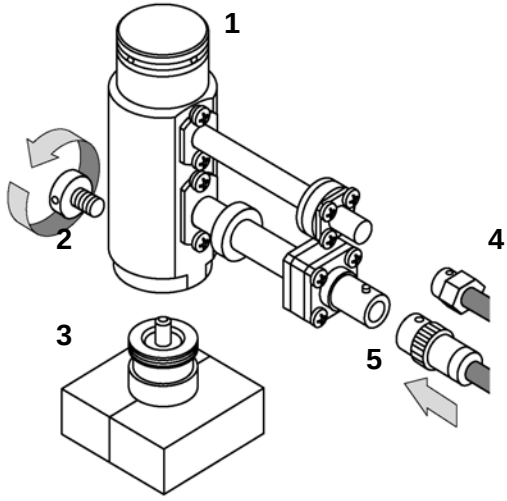
- Jet for NPD
- Tool for jet

1. Place the jet into the detector base body housing and tighten it with the proper tool. Ensure the jet is perfectly vertically aligned to avoid damaging its ceramic part. Refer to Figure 18-2.
2. Install the NPD on the detector base body and secure it by using the fixing screw on the front of the detector cell. Refer to Figure 18-3.
3. Carefully, connect the signal and ignition polarization cables coming from the detector control card, to the detector cell. Refer to Figure 18-3.



1. Tool for jet
2. Jet
3. Detector base body

Figure 18-2. Jet for NPD



1. NPD
2. Fixing screw
3. Detector base body
4. Signal cable
5. Ignition polarization cable

Figure 18-3. Installation of the NPD

NPD Menu

The **DETECTOR (NPD)** menu contains the NPD control parameters. Press LEFT DETECT or RIGHT DETECT to open the **DETECTOR (NPD)** menu. The parameters are explained in Table 18-2.

Table 18-2. Detector (NPD) Menu

Menu	Range	Comments
RIGHT DET (NPD)		This line is the menu title bar.
Source cur, A	On/Off, 1.000–3.500 A in steps of 0.01 A	This is the current applied to heat the thermionic source. It is expressed in amperes. Press ON to turn on the current and to display the setpoint value. Press OFF to turn off the current.
Base temp	On/Off, 0–450 °C	This is the detector base body temperature. Press ON to turn on the heater and to display the actual and setpoint values. Press OFF to turn off the heater and to display the actual value.
Signal pA	Not editable	This parameter shows the collector current in picoamperes (standing current level).
Target curr. pA	3 – 50 pA	This is the target level to be used as a reference value.
Auto adjust	Yes, No	This line indicates the automatic adjustment of the Signal pA to reach the given Target curr pA. Press YES to enable auto adjust.
Polarizer V	1.0–99.0 in steps of 0.1 V	This line indicates the source polarizing voltage in volts.
H2 delay time	On/Off, 0.00–999.9 min	This parameter may be set to interrupt the hydrogen flow during the solvent elution to protect the source. After this time, the hydrogen flow is automatically restored. Press ON to turn on the delay and to display the actual and setpoint values.

Table 18-2. Detector (NPD) Menu (Continued)

Menu	Range	Comments
H2	On/Off, 0–10.0 mL/min in steps of 0.1 mL/min	This line indicates the hydrogen flow supplied to the detector. Press ON to turn on the gas flow and to display the actual and setpoint values. Press OFF to turn off the flow and to display the actual value.
Air	On/Off, 0–600 mL/min	This indicates the air flow supplied to the detector. Press ON to turn on the gas flow and to display the actual and setpoint values. Press OFF to turn off the flow and to display the actual value.
Mkup (N2)	On/Off, 0–100 mL/min	This indicates the make-up gas used with the NPD. The type of the gas is displayed in parentheses. Press ON to turn on the gas flow and to display the actual and setpoint values. Press OFF or 0 to turn off the flow and to display the actual value. The flow remains on when the NPD is off.

OPERATING SEQUENCE

Programming an NPD

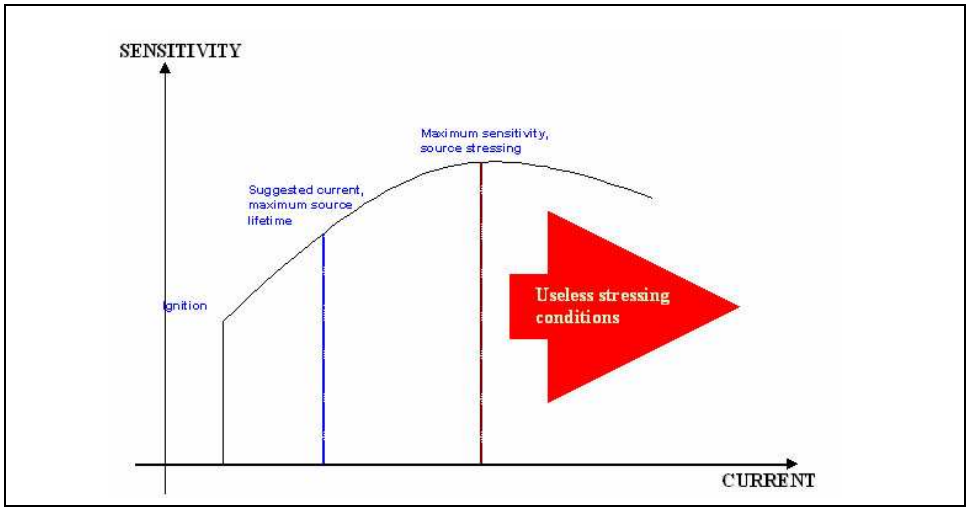
Before you begin this sequence, do the following:

- Verify that all detector gases are connected, a column is correctly installed, and the system is free of leaks.
- Check the oven temperature and injector temperature.
- Check the carrier gas flow according to the capillary or packed column in use.

WARNING! Hydrogen is a potentially dangerous gas. Refer to *Using Hydrogen* on page xxviii for safety information.

1. Press LEFT DETECTOR or RIGHT DETECTOR to open the **`DET (NPD)`** menu.
2. Set the detector `Base temp`.
3. Scroll to `H2` and set the hydrogen flow rate (suggested: 2.3 mL/min).
4. Scroll to `Air` and set the air flow rate (suggested: 60 mL/min).
5. Scroll to `Mkup` and set the make-up flow (suggested: 15 mL/min).
6. Scroll to `Polarizer V` and set 3.5 V.
7. Scroll to `Source cur,` and set the heating current value. Wait for a few seconds and verify the ignition of the gas layer around the thermionic source.

NOTE The suggested heating current is the minimum one, able to give enough sensitivity. Sensitivity is not just corresponding to the absolute peak intensity but it is related to the signal-to-noise ratio. A decline of the peak intensity during time is normal for a thermionic source and doesn't necessarily correspond to a loss of sensitivity. In case that a higher sensitivity is required, a slight increase of the source current can be applied. Do not exceed with the source current. In fact, as showed in the picture below, initially the sensitivity increases with the source current, but at too high currents the sensitivity can even decrease, although the peak intensity is higher (this is due to higher noise level). Operate at too high currents is a stressing condition for the NPD source and may jeopardize source life time.



CAUTION As a general rule THE HIGHER IS THE SOURCE CURRENT AND THE SHORTER IS THE SOURCE LIFETIME.

To turn on the source the first time, follow the sequent steps:

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, 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 stabilizes.

To check that source is correctly switched on, please proceed as follows:

— Decrease hydrogen flow to 0.5 mL/min until signal decreases down to zero, then increase again to original value.

- 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.

- If the signal rises back to original value, it means that source is correctly switched on

d. Increase the current value of 2% of the actual ignition current. Let the signal stabilizes until its level drops below 20 pA.

WARNING! Changes of gas flows and of detector base temperature affect the source current value required.

8. Press LEFT SIGNAL or RIGHT SIGNAL to open the detector **`SIGNAL (NPD)`** menu and verify the output signal.

Refer to the *Setting the NPD Signal Parameters* operating sequence on page 387 for instructions.

OPERATING SEQUENCE

Setting the NPD Signal Parameters

1. Press LEFT SIGNAL or RIGHT SIGNAL to enter the detector **`SIGNAL (NPD)`** menu.
2. Scroll to `Range 10^ (0...3)` and select the electrometer amplifier input range. `0` (10^0) is the most sensitive.
3. If output signal filtering is required, scroll to `Analog filter` and press ON.
4. Scroll to `Auto zero?` and press ON.
5. If offset is required, scroll to `Offset` and enter a numeric value or press ON to recall the last offset from memory.
6. Turn `Baseline comp` ON if you want to compensate the baseline.

NOTE

If the `Range 10^` is set 2 or 3, the small variation of the output signal is not detected. For this reason the `Signal pA` parameter will be not displayed in the **`DETECTOR NPD`** menu.

19

Photoionization Detector (PID)

This chapter describes the operating sequences and principles for the Photoionization Detector (PID).

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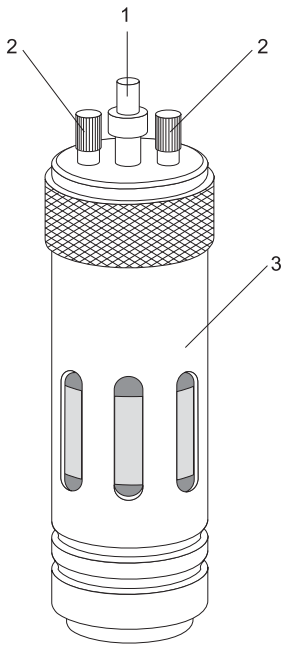
PID Gas Supplies 393
Detectors Coupled in Series to the PID 395
PID Installation 397
PID Menu 403

Operating Sequences

Programming a PID 404
Setting the PID Signal Parameters 405
Shutting Down the PID 406

PID Overview

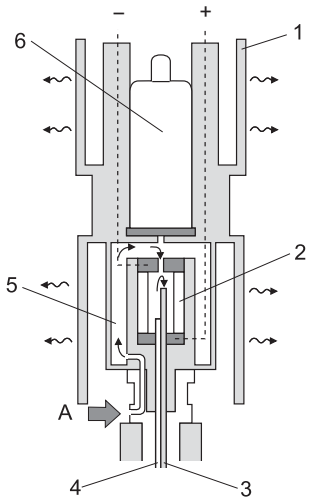
The PID detection unit, shown in Figure 19-1, consists of a hot cell assembly surrounded by a stainless steel bell. The bell guides the gas that thermally insulates the cell from the lamp housing. It also purges the external side of the cell to prevent air from diffusing into the cell.



1. UV Lamp Holder
2. Knurled Screws
3. Detector Heat Sink

Figure 19-1. The Photoionization Detector

All the gases (carrier, make-up, and sheath gas) leave the detector through the exit tube as shown in Figure 19-2.



1. Heat Sink
2. Quartz Cell
3. Exit Line
4. Column
5. Gas Insulation
6. UV Lamp

A. Sheath Gas

Figure 19-2. PID (Cutaway View)

The lamp housing, located above the bell, contains all the electrical contacts and acts as a support for the lamp holder. The UV lamp inside the lamp holder is easily removable for replacement operations.

The lamp housing and UV lamp are kept at a low temperature (<100 ºC) by a heat sink which dissipates the heat of the detector base body by convection.

The detector cell consists of an all-quartz ionization chamber containing two electrodes (polarizing and collecting), to which a voltage of 300 V is applied.

The ionization chamber is the hot part of the detector. The UV lamp, the sealed window, and the lamp housing are kept relatively cold by the heat sink and sheath gas.

Operating Principles

The PID operates on the principle of absorption of energy (photons) emitted by an UV lamp by sample molecules. This leads to an ionization process described in the following equation:

$$AB + h\nu \Rightarrow AB^{+} + e^{-}$$

This process occurs when the molecules have ionization potential less than or roughly equal to the energy of the UV lamp used. The use of different lamps makes it possible to achieve different detection selectivity. As a general rule, the lamp emitting the lowest energy photons provides the highest selectivity.

Appendix A, *Ionization Potential of Selected Molecules*, contains information to help you determine the lamp intensity necessary to ionize several different types of molecules.

PID Applications

The PID is mainly used to determine aromatic pollutant compounds in environmental applications and to analyze polycyclic aromatic hydrocarbons. In addition, this detector may also be used to determine alkenes and some inorganic substances such as arsine, phosphine, and ammonia. The PID performance is better than that of the TCD in terms of sensitivity and selectivity for these substances.

To prevent memory effects and contamination with the sample, operate the PID at temperatures higher than 300 ºC. It can be baked-out at temperatures of up to 400 ºC. Due to its innovative thermal design, the lamp lifetime is not reduced at such high temperatures.

UV Lamp Types

Four easily interchangeable UV lamps are available for analyzing different compounds. Table 19-1 shows the different lamps and their applications. Refer to Appendix A, *Ionization Potential of Selected Molecules*, to determine the lamp intensity necessary for your application.

Table 19-1. PID UV Lamps

Lamp Type	Application
8.4 eV	This lamp is used for the determination of amines and polycyclic aromatic compounds. It provides the highest selectivity.
9.6 eV	This lamp is used for specific determination of low boiling aromatic compounds (BTEX analyses).
10.6 = 10.0 (10.2) eV	This lamp is used for general applications.
11.8 eV	This lamp is used for the determination of aldehydes and ketones.

NOTE All the UV lamps currently on the market that have 10.0 or 10.6 labels are identical. They contain krypton gas which emits both 10.0 and 10.6 eV radiations. The krypton-filled lamp also qualifies as a 10.2 eV lamp.

Life of the 11.8 eV Lamp

The expected life time of a PID lamp depends on how the lamp is operated. High temperature, high current through the lamp, window cleanliness are all factors that can deteriorate the lamp emission. The 11.8 eV lamp is constructed with a Lithium Fluoride window that is needed to transmit energies of 11.7 eV and higher. The Lithium Fluoride is especially subjected to alterations by the UV light, emitted by the lamp itself, water vapor and high temperatures. This deterioration can be clearly noticed when the Lithium Fluoride window becomes yellow. To reduce the deterioration of your 11.8 eV lamp we suggest to use the lowest PID temperature, compatible with your analytical method.
When installing a new 11.8 eV PID lamp, an initial steep response decrease must be expected.

PID Gas Supplies

The PID requires three gas flows:

- carrier gas

- make-up gas
- sheath (purge) gas

The following gases can be used for the PID carrier gas supply:

- helium (preferred)
- nitrogen
- hydrogen (for capillary columns)

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 following gases can be used for the PID make-up gas supply:

- helium (preferred)
- nitrogen

NOTE

The make-up gas you use also depends on the type of detector used in series with the PID, if any. Refer to *Detectors Coupled in Series to the PID* on page 395 for more information.

The following gases can be used for the PID sheath gas:

- helium (for detector temperature up to 300 ºC)
- nitrogen (for detector temperature over 300 ºC)

Flow Rates

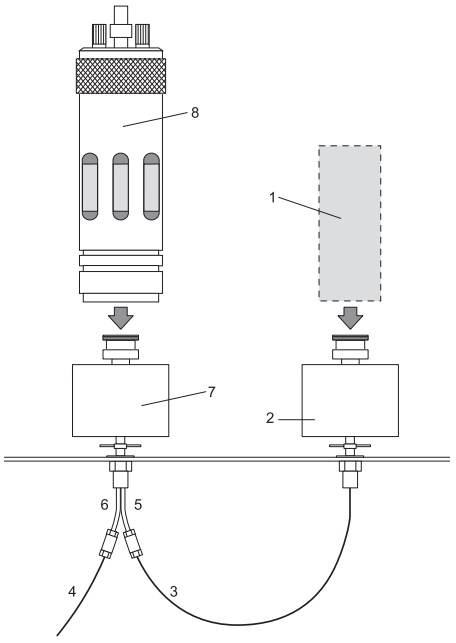
The following gas flow rates are recommended for the PID:

- make-up gas: 5–10 mL/min
- sheath gas: 30–40 mL/min

To obtain the maximum sensitivity and resolution, the total flow rate of carrier and make-up gas together should be 8–10 mL/min.

Detectors Coupled in Series to the PID

You can couple another detector in series to the PID by connecting the outlet of the exit line to the second detector base body, as shown in Figure 19-3.



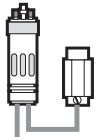
1. Auxiliary Detector	5. Exit Line
2. 2nd Detector Base Body	6. Inlet Line
3. Fused Silica Tubing	7. Detector Base Body
4. Column	8. PID

Figure 19-3. PID/Second Detector Coupling

The make-up and purge gas flow through the exit line. The addition of other gases is not usually necessary.

The sheath gas should be nitrogen or helium, depending on the requirements of the detector coupled in series to the PID.

PID/FID Configuration



The PID/FID coupling is the most common arrangement. The FID makes troubleshooting easier and more indicative.

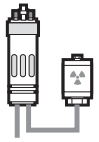
Use a selective UV lamp (8.4–9.6 eV) in the PID because the FID provides a universal response.

The required gases are as follows:

- carrier gas—helium, nitrogen, or hydrogen
- make-up gas—helium or nitrogen
- sheath gas—nitrogen or helium

The flow of hydrogen for the FID should be slightly increased to improve flame stability and to prevent the flame from extinguishing due to sample overload. Refer to *FID Gas Supplies* on page 357 for more information.

PID/ECD Configuration



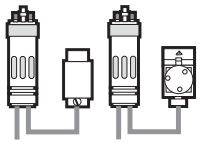
The PID/ECD coupling is helpful for environmental analyses to obtain more analytical information in a single run.

The required gases are as follows:

- carrier gas—helium, nitrogen, or hydrogen
- make-up gas—nitrogen
- sheath gas—nitrogen

The PID sheath gas also provides make-up gas for the ECD. Decrease the ECD make-up gas flow accordingly. Refer to *ECD Gas Supplies* on page 367 for more information

PID/NPD or FPD Configuration



These configurations allow nitrogen/phosphorous and sulphur/phosphorous heterocompounds to be selectively detected in addition to the PID response.

The required gases are as follows:

- carrier gas—helium, nitrogen, or hydrogen (with some limitations)
- make-up gas—helium or nitrogen
- sheath gas—helium or nitrogen

NOTE The make-up and purge gas total flow can affect the NPD response. No relevant influence is produced on the FPD response.

PID Installation

This operation allows the correct installation of the PID on your TRACE GC Ultra.

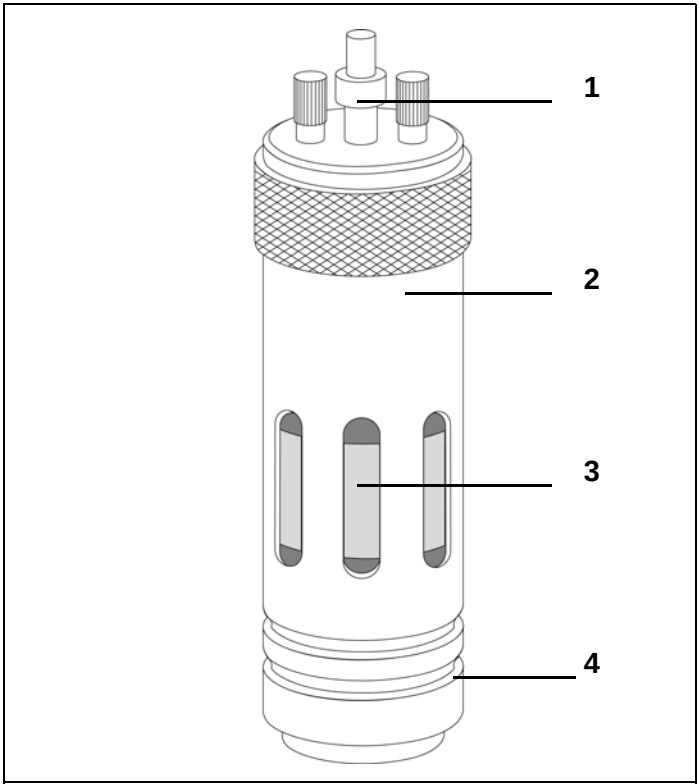
Material required

- UV Lamp
- Fixing Tool

The PID consists of four main sub units. Refer to Figures 19-4 and 19-5 to identify the parts constituting the PID.

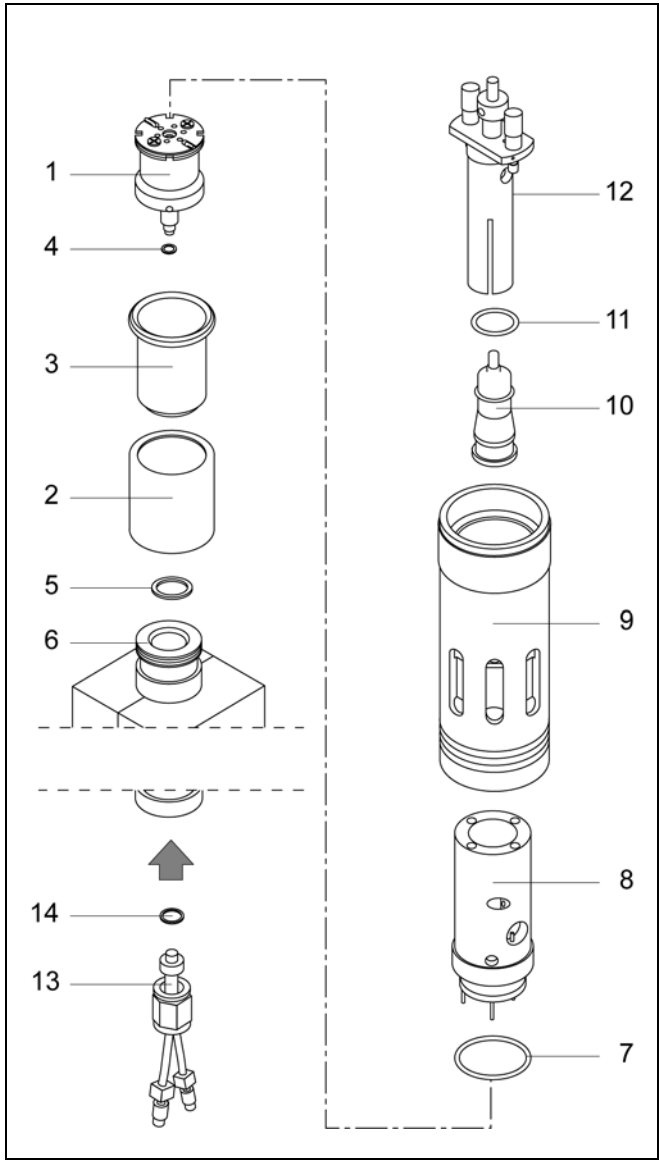
- *Cell Block*
  It includes the detector cell assembly, the stainless steel bell and the insulation jacket.
- *Lamp Housing*
  It includes the detector cell assembly, the stainless steel bell and the insulation jacket.
- *Lamp Holder*
  It contains the UV lamp with the electrical cable for lamp ignition and operation.

- *Heat Sink*
  It dissipates the heat of the detector base body.



1. Lamp Holder	2. Heat Sink
3. Lamp Housing	4. Cell Block (not visible)

Figure 19-4. PID General View



1. Detector Cell Assembly
2. Insulation Jacket
3. Stainless Steel Bell
4. Seal
5. Seal
6. Detector Base Body
7. Viton™ O-ring for Lamp Holder
8. Lamp Housing
9. Heat Sink
10. UV Lamp
11. Viton™ O-ring for UV Lamp
12. Lamp Holder
13. Two-way Capillary Adapter
14. Silver Seal
15. Fixing Tool

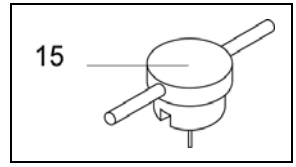


Figure 19-5. Explose of the PID Components

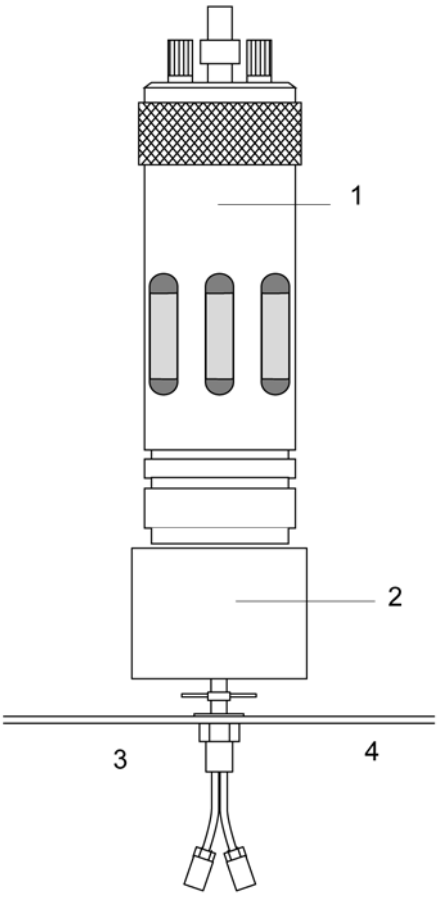
To install the PID on the GC detector base body, follow the instruction below: Refer to Figure 19-5 to identify the parts.

1. Place the insulation jacket (**2**) on the stainless steel bell (**3**).
2. Put the detector cell assembly (**1**) into the stainless steel bell (**3**) passing the lower threaded section of the cell assembly through the bottom hole of the bell.
3. Install the seal (**5**) on the detector base body surface (**6**) and the seal (**4**) on the threaded section of the cell that goes out from the hole of the bell.
4. Screw the cell block (detector cell assembly + stainless steel bell + insulation jacket) on the detector base body, without overtighten, by using the fixing tool (**15**) provided.
5. Make sure that the Viton™ O-ring (**8**) is correctly positioned on the lower part of the lamp housing (**7**).
6. Pull the electrical cables of the lamp housing (**7**) through the heat sink (**9**) pay attention that the external knurled area of the heat sink is oriented upwards and the internal threaded section must be turned towards the detector base body.
7. Put the lamp housing on the cell block paying attention to the proper insertion of the two orientation pins into the corresponding slots of the cell block.
8. Mount the heat sink (**9**) on the lamp housing (**7**), then screw manually the heat sink on the stainless steel bell.
9. Install the UV lamp (**10**), with the Viton ™ O-ring (**11**) on its flange, into the lamp holder (**12**).

WARNING! Never install the UV lamp without the o-ring.

10. Install the lamp assembly (UV lamp + lamp holder) into the lamp housing and ensure screwing the two knurled screws. Refer to the TRACE GC Ultra *Maintenance and Troubleshooting Manual*.

11. Mount the two-way capillary adapter (**13**) to the lower part of the detector base body, inside the GC column oven, interposing the seal (**14**).
The result of the operation is shown in Figure 19-6.



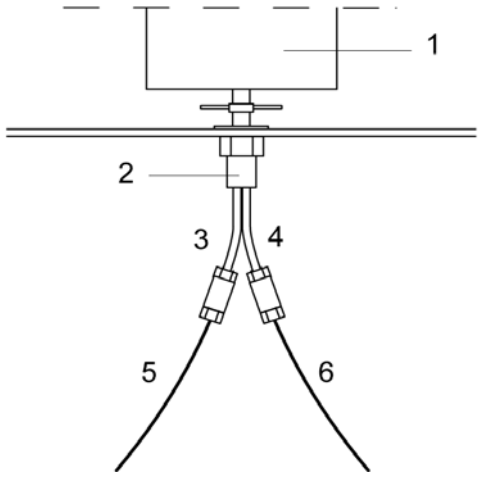
1. PID Assembly	2. Detector Base Body
3. Two-way Capillary Adapter	4. GC Column Oven

Figure 19-6. PID Installation Result

Connecting Capillary Column and Exit Line

WARNING! Before connecting capillary column and exit line, perform the detector leak test as described in the *TRACE GC Ultra Maintenance and Troubleshooting Manual*.

12. Connect capillary column and exit line to the PID as described in *Chapter* 14 on page 298. The result of the operation is shown in Figure 19-7.



1. Detector Base Body	2. Two-way Capillary Adapter
3. Inlet Line	4. Exit Line
5. Capillary Column	6. Exit Line Fused Silica Tubing

Figure 19-7. Capillary Column and Exit Line Connections

PID Menu

The **DET (PID)** menu contains the PID control parameters. Press LEFT DETECTOR or RIGHT DETECTOR to open the menu shown in Table 19-2.

Table 19-2. Detector (PID) Menu

Menu	Range	Comments
Right Det (PID)		This is the menu title bar.
Lamp	On/Off	This parameter indicates the UV lamp status. Press ON to turn on the lamp. Press OFF to turn it off.
Base temp	On/Off, 30–450 ºC	This is the detector base body temperature. Press ON to enable the heater and display the actual and setpoint values. Press OFF to turn off the heater and display the actual value.
High current mode?	No (1 mA) Yes (2 mA)	This indicates the type of current applied to the UV lamp. Press YES to select a high current.
Signal pA	Not editable	This parameter shows the standing current level in picoamperes.
Mkup (N2)	On/Off, 0–100 mL/min	This parameter indicates the make-up gas used with the PID. The type of gas is displayed in parentheses. Press ON to turn on the make-up gas flow and display the actual and setpoint values. Press OFF to turn off the flow and display the actual value.
Sheath gas	On/Off, 0–99 mL/min.	This parameter indicates the sheath gas used with the PID. Press ON to turn on the sheath gas flow and display the actual and setpoint values. Press OFF to turn off the flow and display the actual value.

OPERATING SEQUENCE

Programming a PID

Before you begin, do the following:

- Verify that all detector gases are connected, a column is correctly installed, and the system is free of leaks.
- Verify the electrical connections.
- Check the oven temperature and injector temperature.
- Check the carrier gas flow according to the capillary column in use.

WARNING! Hydrogen is a potentially dangerous gas. Refer to *Using Hydrogen* on page xxviii for hydrogen safety information when using hydrogen as a carrier gas.

1. Press LEFT DETECT or RIGHT DETECT to open the **`DET (PID)`** menu.
2. If the detector requires conditioning, scroll to `Base temp` and set the detector base body temperature to 350 ºC for 2–3 hours. Then set the temperature at the operating value for the analytical requirements.
3. Scroll to `Mkup` and change the make-up gas flow rate, if necessary.
4. Scroll to `Sheath gas` and change the sheath gas flow rate, if necessary.
5. Scroll to `High current mode?` and press ON to select a high current, if desired.
6. Scroll to `Lamp` and press ON. This starts the UV lamp ignition. A sudden baseline deflection will also indicate that the lamp is lit inside the detector.

   A `Lamp failure` message is displayed if the UV lamp is not lit. Refer to the *Maintenance and Troubleshooting Manual* for more information.
7. Press LEFT SIGNAL or RIGHT SIGNAL to open the detector **`SIGNAL (PID)`** menu and verify the output signal.

Refer to the *Setting the PID Signal Parameters* operating sequence on page 405 for more information.

After you enter the correct parameters, the PID requires a short period of conditioning to obtain a stable baseline.

To extend the lamp lifetime, turn off the UV lamp when the detector is not being used for extended periods of time (for example, overnight or on weekends). Refer to the *Shutting Down the PID* operating sequence on page 406 for more information.

NOTE

The detector base body temperature, the total gas flow, and the lamp current influence the background level as well as signal and noise. The optimal values can be determined experimentally.

OPERATING SEQUENCE

Setting the PID Signal Parameters

1. Press LEFT SIGNAL or RIGHT SIGNAL to enter the detector **`SIGNAL (PID)`** menu.
2. Scroll to `Range 10^ (0...3)` and select the electrometer amplifier input range. 0 (10^0) is the most sensitive.
3. If output signal filtering is required, scroll to `Analog filter` and press ON.
4. Scroll to `Auto zero?` and press ON.
5. If offset is required, scroll to `Offset` and enter a numeric value or press ON to recall the last offset from memory.
6. Turn `Baseline comp` ON if you want to compensate the baseline.

NOTE

If the `Range 10^` is set 2 or 3, the small variation of the output signal is not detected. For this reason the `Signal pA` parameter will be not displayed in the **`DETECTOR PID`** menu.

OPERATING SEQUENCE

Shutting Down the PID

Overnight

To shut down the PID overnight, use the following sequence:

1. Press LEFT DETECTOR or RIGHT DETECTOR to open the **DET (PID)** menu.
2. Scroll to Lamp and press OFF to turn the UV lamp off.
3. Reduce the gas flows, if desired.

The operating temperature should remain unchanged.

Weekends

To shutdown the PID on weekends, use the following sequence:

1. Press LEFT DETECTOR or RIGHT DETECTOR to open the **DET (PID)** menu.
2. Scroll to Lamp and press OFF to turn the UV lamp off.
3. Reduce the gas flows, if desired.

The operating temperature should be reduced below 300 ºC.

Long Period and/or Cell Maintenance

To shutdown the PID for an extended period of time or for the maintenance of the cell, use the following sequence:

1. Press LEFT DETECTOR or RIGHT DETECTOR to open the **DET (PID)** menu.
2. Scroll to Lamp and press OFF to turn the UV lamp off.
3. Reduce the temperature of the detector base body to 60–80 ºC.
4. Turn off all gas flows when the temperature is below 100 ºC.

20

Flame Photometric Detector (FPD)

This chapter describes the operating principles and sequences for the Flame Photometric Detector (FPD).

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FPD Gas Supplies 411
FPD Installation 413
FPD Menu 415

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Programming an FPD 417
Setting the FPD Signal Parameters 419

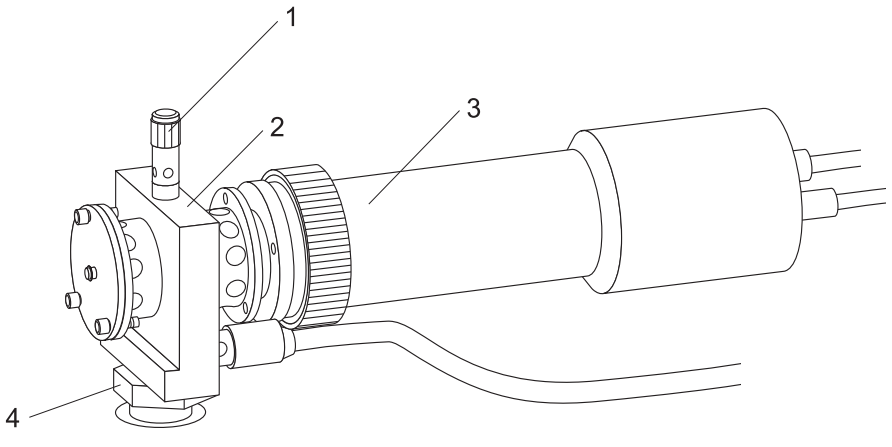
FPD Overview

The FPD, shown in Figure 20-1, is based on the measurement of the characteristic radiation emitted by particular 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 of 394 nm while HPO* has a maximum emission of 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.

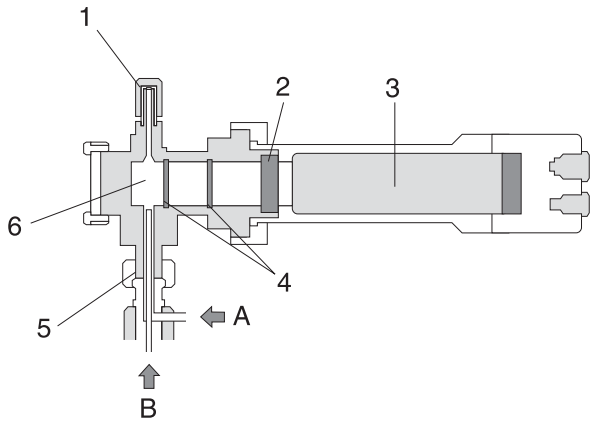


1. Combustion Gas Exit/Light Baffle
2. Detector Body
3. Photomultiplier Tube
4. Fixing Nut

Figure 20-1. Flame Photometric Detector

FPD Description

The FPD detector consists of a combustion chamber, a narrow band interferential filter, and a photomultiplier tube for measuring the chemiluminescent emission. Figure 20-2 shows the body of the detector, including the special burner, the heater and the temperature sensor, the flame ignitor, and the heat shields connected to the photomultiplier tube. The exhaust gases and the combustion products are vented through the combustion gas exit. The detector is equipped with both the sulphur filter (focused at 394 nm) and the phosphorous filter (focused at 526 nm).

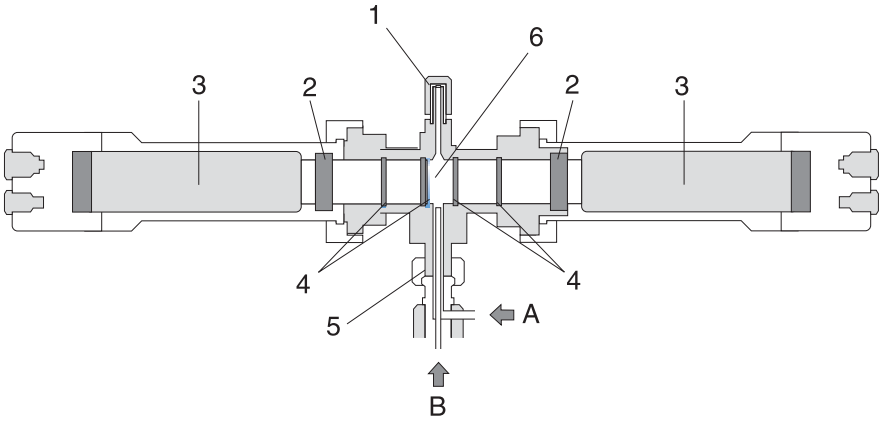


1. Combustion Gas Exit/Light Baffle	5. Fixing Nut
2. Interferential Filter	6. Detector Cell
3. Photomultiplier Tube	A. Air inlet
4. Heat Shields	B. Effluent from the column + hydrogen

Figure 20-2. FPD Cutaway View

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. This configuration allows to process a sample for phosphorous and sulphur profile simultaneously, or phosphorous and tin with suitable interferential filter (610 nm). Figure 20-2 shows the Dual FPD detector.



1. Combustion Gas Exit/Light Baffle	5. Fixing Nut
2. Interferential Filter	6. Detector Cell
3. Photomultiplier Tube	A. Air inlet
4. Heat Shields	B. Effluent from the column + hydrogen

Figure 20-3. Dual FPD Cutaway View

To perform Dual FPD detector configuration, the appropriate upgrade kit is required. The second photomultiplier tube must be configured as **auxiliary** detector.

Jet

The metal jet is mounted on the detector base body for capillary and wide-bore (CB 71) or packed columns (CB 70).

FPD Heating

The temperature should 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 ºC) and raise the base body temperature to a higher value (280–350 ºC) depending on the analytical requirements. Higher detector temperatures (300 ºC–350 ºC) could be used for ECD/FPD tandem configuration when required.

FPD Gas Supplies

The carrier gases normally used with the FPD are shown in Table 20-1.

Table 20-1. FPD Carrier Gases

Carrier Gas	Capillary Columns	Packed Columns
helium	X	X
nitrogen	X	X
hydrogen	X	---
argon	---	X

The carrier gas flow range depends on the type of gas used and on the type and diameter of the capillary or packed column installed.

The detector fuel gases used with the FPD are:

- hydrogen
- air

Make-up gas is generally not required with the FPD.

The right choice of hydrogen/air flow rates is of primary importance in FPD sensitivity and selectivity. Suggested flow rates are listed in Table 20-2.

Table 20-2. Suggested FPD Gas Flow Rates

Gas	Capillary Column	Packed Column
carrier	1–3 mL/min	30–50 mL/min
hydrogen	85–100 mL/min	100–120 mL/min
air	100–120 mL/min	110–135 mL/min

The optimum air flow rate should be determined experimentally by analyzing a standard mixture after correctly setting the hydrogen flow rate.

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 120 to 90 mL/min), while maintaining the same hydrogen flow rate. This possibility can be especially useful in the analysis of organophosphorous pesticide residues.

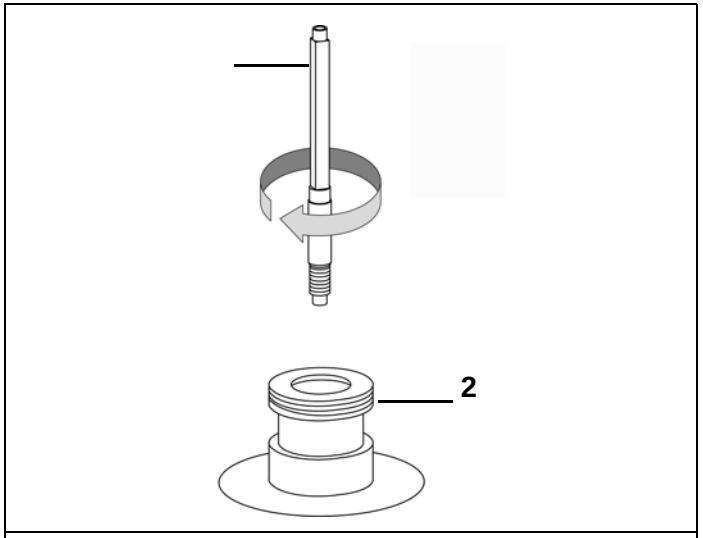
FPD Installation

This operation allows the correct installation of the FPD on your TRACE GC Ultra.

Material required

- Jet for FPD
- 5-mm wrench
- FPD fixing tool.

1. Place the jet into the detector base body housing and tighten it. Ensure the jet is perfectly vertically aligned to avoid damage.

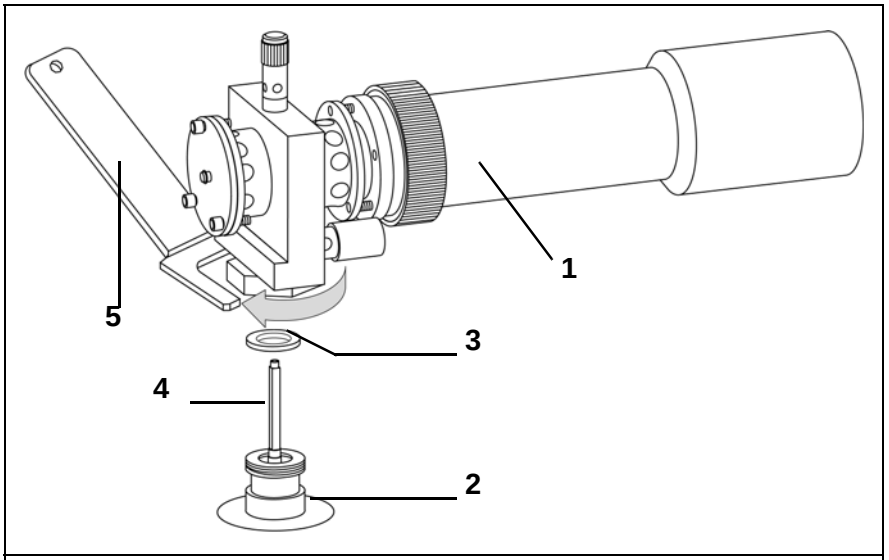


1. Jet 2. Detector Base Body

Figure 20-4. Jet for FPD

2. Place the FPD on the detector base body, paying attention that the aluminium ring has been inserted in the correct position.

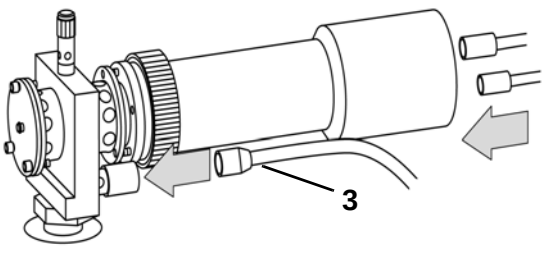
3. Tighten the fixing nut by using the FPD fixing tool.



1. FPD	2. Detector Base Body
3. Aluminium ring	4. Jet
5. FPD Fixing Tool	

Figure 20-5. Installation of the FPD

4. Carefully, connect the signal, excitation voltage and ignition/heating cables coming from the detector control card, to the detector cell.



1. Signal Cable	2. Excitation Voltage Cable
3. Ignition/heating Cable	

Figure 20-6. Cables Connection

FPD Menu

The **DET(FPD)** menu contains the FPD control parameters.
Press LEFT DETECT or RIGHT DETECT to open the menu shown in Table 20-3.

Table 20-3. Detector (FPD) Menu

Menu	Range	Comment
RIGHT DET (FPD)		This line is the menu title bar.
Flame	On/Off	This line indicates the flame status. Press ON to turn on the air flow and ignitor and turn on H_2 for ignition. On is displayed if the temperature is ≥ 120 ºC. If not, an error message is displayed.
Base temp	On/Off, 30–450 ºC	This is the detector base body temperature. Press ON to turn on the heater and display setpoint and actual values. Press OFF to turn off the heater and display the actual value.
FPD temp	On/Off, 30–350 ºC	This is the detector temperature. Press ON to turn on the heater and display the actual and setpoint values. Press OFF to turn off the heater and display the actual value.
Signal pA	Not editable	This parameter shows the standing current level in picoamperes. The displayed value also indicates the flame status.
High voltage mode	Yes (900 V) No (800 V)	This parameter indicates the value of voltage applied to the photomultiplier tube. Press ON to select high voltage.
H2	On/Off, 0–200 mL/min	This line indicates the hydrogen flow to the detector. Press ON to turn on the H_2 flow and display the actual and setpoint values. Press OFF or 0 to turn off the flow. This flow can be turned on independently when the FPD is off, but it cuts off automatically when the FPD is turned from On to Off.

Table 20-3. Detector (FPD) Menu (Continued)

Menu	Range	Comment
Air	On/Off, 0–600 mL/min	This parameter indicates the air flow to the detector. Press ON to turn on the air flow and display the actual and setpoint values. Press OFF or 0 to turn off the flow. This flow can be turned on independently when the FPD is off, but it cuts off automatically when the FPD is turned from On to Off.
Mkup (N2)	On/Off, 0–100 mL/min	This line indicates the make-up gas flow to the detector. Press ON to turn on the flow and display the actual and setpoint values. Press OFF or 0 to turn off the flow.

Dual FPD Menu

When the second photomultiplier tube is connected to the FPD detector and configured as auxiliary detector, the control parameters are contained in the **AUX DETECTOR** menu.
Press AUX, then scroll to Detector and press ENTER to open the menu shown in Table 20-3.

Table 20-4. Dual FPD Menu

Menu	Range	Comment
AUX DETECT (DualFPD)		This line is the menu title bar.
Signal pA	Not editable	This parameter shows the standing current level in picoamperes. The displayed value also indicates the flame status.
High voltage mode	Yes (900 V) No (800 V)	This parameter indicates the value of voltage applied to the second photomultiplier tube. Press ON to select high voltage.

OPERATING SEQUENCE

Programming an FPD

Before you begin, do the following:

- Verify that all detector gases are connected, a column is correctly installed, and the system is free of leaks.
- Check the oven temperature and injector temperature.
- Check the carrier gas flow according to the capillary or packed column in use.

WARNING! Hydrogen is a potentially dangerous gas. Refer to *Using Hydrogen* on page xxviii for hydrogen safety information when using hydrogen as a carrier gas.

1. Press LEFT DETECT or RIGHT DETECT to open the **`DET (FPD)`** menu.
2. Scroll to `Base temp` and set the detector base body temperature according to the analytical requirement.
3. Scroll to `FPD temp` and set the detector temperature. This must be greater than 120 ºC to avoid water condensation on the heat shields.
4. Scroll to `H2` and enter the correct hydrogen flow.
5. Scroll to `Air` and enter the correct air flow rate.
6. Scroll to `Mkup` and enter a make-up gas flow rate, if required, or press OFF.
7. Scroll to `High voltage mode?` and press ON if high voltage is required.
8. Scroll to `Flame` and press ON. This starts the ignition sequence.

Positive variation of the Signal pA 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.

9. Press LEFT SIGNAL or RIGHT SIGNAL to open the detector **`SIGNAL (FPD)`** menu and verify the output signal.

Refer to the *Setting the FPD Signal Parameters* operating sequence on page 419 for more information.

Programming the Dual FPD Parameter

1. Press AUX , then scroll to `Detector` and press ENTER to open the **`AUX DETECT(DualFPD)`** menu.

2. Scroll to `High voltage mode?` and press ON if high voltage is required.

Observe the variation of the Signal pA value

1. Press AUX , then scroll to `Signal` and press ENTER to open the **`AUX SIGNAL (DualFPD)`** menu and verify the output signal.

Refer to the *Setting the FPD Signal Parameters* operating sequence on page 419 for more information.

OPERATING SEQUENCE

Setting the FPD Signal Parameters

1. Press LEFT SIGNAL or RIGHT SIGNAL to enter the detector **`SIGNAL (FPD)`** menu:
2. Scroll to `Range 10^ (0...2)` and select the electrometer amplifier input range. 0 (10^0) is the most sensitive.
3. Scroll to `Auto zero?` and press ON.
4. If offset is required, scroll to `Offset` and enter a numeric value or press ON to recall the last offset from memory.
5. Turn `Baseline comp` ON if you want to compensate the baseline.

Dual FPD Signal Parameters

1. Press AUX , then scroll to `Signal` and press ENTER to open the **`AUX SIGNAL (DualFPD)`** menu.
2. Scroll to `Range 10^ (0...2)` and select the electrometer amplifier input range. 0 (10^0) is the most sensitive.
3. Scroll to `Auto zero?` and press ON.
4. If offset is required, scroll to `Offset` and enter a numeric value or press ON to recall the last offset from memory.
5. Turn `Baseline comp` ON if you want to compensate the baseline.

If the `Range 10^` is set 2, the small variation of the output signal is not detected. For this reason the `Signal pA` parameter will be not displayed in the **`DETECTOR FPD`** menu.

21

Thermal Conductivity Detector (TCD)

This chapter describes the operating principles and sequences for the Thermal Conductivity Detector (TCD).

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TCD Overview

The TCD is sensitive to any compound having thermal conductivity other than that of the carrier gas used. The TCD is a universal type detector. It has a broad range of uses in the analysis of permanent gases and other organic or inorganic compounds for which the Flame Ionization Detector (FID) is practically non-sensitive, such as CO_2, CS_2, H_2O, H_2, and N_2.

While the FID is more sensitive to most organics, the simplicity of the TCD often makes it the preferred detector when analyte concentrations are high enough. The TCD typically requires only one type of gas, such as helium. The FID requires up to four.

Because the TCD is a non-destructive detector, it can be connected in series to other chromatographic detectors.

The TCD consists of a stainless steel block containing two filaments (generally tungsten/rhenium filaments) which have the same electrical resistance. The block is housed in an aluminum case that accommodates the heating elements and the temperature sensor.

NOTE TCD with polyimide coated filaments is optionally available for the analysis of very aggressive gas matrices.

The filaments are electrically connected to a Wheatstone bridge. Two gas flows, a reference flow and an analytical flow, enter the TCD cell, pass across the filaments, and vent to the atmosphere. Figure 21-1 shows the filaments and gas flows.

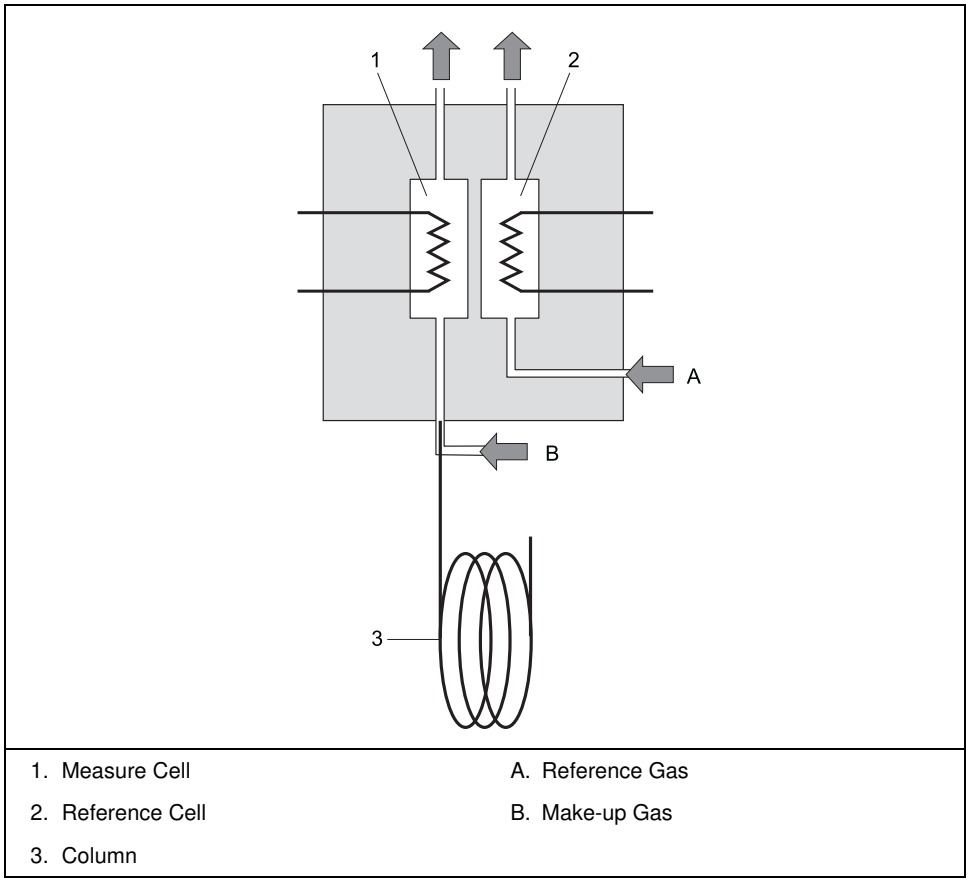


Figure 21-1. TCD Filaments and Gas Flows

When the filaments are properly powered, they heat at a temperature (resistance) that is a function of the thermal conductivity of the gas flowing through the filaments. When a chromatographic component elutes in the analytical channel, a change takes place in the heat transfer followed by a variation of the filament temperature.

The output signal is sent to a Thermo Scientific data system software. The signal polarity is a function of the thermal conductivity of the component relative to the reference gas and to the user-selected polarity of the filament power supply.

WARNING! The TCD filaments are sensitive to impurities present in the carrier, reference, and make-up gas supplies. To ensure correct detector operation, you should use oxygen and water vapor traps in the carrier gas and the make-up gas supply lines. We suggest that you install an OXICLEAR filter (PN 281 131 40) before connecting the gas to the GC.

TCD Gas Supplies

The TCD detector requires the same gas whether for the measure channel (carrie and make-up gas, when necessary) and the reference channel (reference gas).

Helium is the recommended carrier gas due to its high thermal conductivity and chemical inertness. Low conductivity gases (argon, nitrogen) are used for special analytical requirements.
With special precautions, you can also use hydrogen as the carrier and detector gas.

WARNING! Hydrogen is a potentially dangerous gas. Refer to *Using Hydrogen* on page xxviii for safety information.

Table 21-1 contains information about the thermal conductivity of several gases.

Table 21-1. Thermal Conductivity of Gases

Gas	Thermal Conductivity ($\lambda x 10^7$) at 0 ºC where λ=Cal/cm x sec. x ºC
Hydrogen	4130
Helium	3363
Methane	720
Oxygen	583
Nitrogen	580
Carbon Oxide	540
Argon	406
Carbon Dioxide	343

Columns

The TCD requires two separate gas lines. One connects to the analytical column and the other connects to the reference channel. The reference channel connects to the DGFC module. This eliminates the need for a second column. The column effluent connects to the analytical cell along with the make-up gas, if required, from the DGFC detector module. Should the use of wide-bore or capillary columns be required, the connection between the column and injector must necessarily be modified. When using capillary columns, the make-up line must be activated. This line has to feed the analytical channel at the column outlet, thus compensating the special flows required by capillary columns. For column installation instructions, refer to Chapter 14, *Columns*.

TCD Operating Modes

The TCD can operate in constant temperature or constant voltage mode. It can also automatically switch to a *constant current mode* when the filaments reach the maximum allowable current value of 125 mA.

Constant Temperature

In constant temperature mode, the filament temperature remains constant at a set value. A feedback loop circuit changes the voltage as the gas thermal conductivity changes. If the required voltage reaches the maximum allowable value of 15 V, the system will automatically switch to the *constant voltage mode*.

Constant Voltage

In constant voltage mode, the filament voltage remains constant at a set value. The temperature variation, positive or negative, generates a current variation, negative or positive, that will give the corresponding signal. The voltage values range from 5 to 15 V. If the current reaches the maximum allowable value of 125 mA, the system will automatically switch to the *constant current mode*.
When the *constant voltage mode* is used, it is necessary to set the filament temperature limit in the TCD detector menu (Table 21-4). If this value is reached, the system will automatically switch to the *constant temperature mode*.

Automatic Switching of Control Options

The automatic switch function is always active. It allows automatic switching from one operating mode to another depending on the parameters set and the carrier gas used.

Automatic Switching From Constant Voltage to Constant Temperature

The following is an example of TCD operating conditions:

- carrier gas: helium (high thermal conductivity)
- cell temperature: 100 ºC
- constant voltage: 10 V
- filament temperature limit: 200 ºC

In constant voltage mode of 10 V with a 200 ºC filament temperature limit, when a compound of a particular thermoconductivity enters the cell, it causes the filament temperature to increase. When the filament temperature reaches the filament temperature limit, the system automatically switches to constant temperature mode and the voltage changes.

Automatic Switching to Constant Current Mode

Every time the set values of filaments voltage, block temperature and filaments temperature cause the filament current to reach the maximum value of 125 mA, the system will automatically switch to the *constant current mode* and the filaments cannot be heated more than the correspondent temperature.
This mode has good sensitivity and a linearity comparable to that obtained with the CV mode. However, the high filament temperatures can potentially shorten the filament life.

NOTE The constant current mode operates only when using high thermal conductivity gases, such as helium.

Selecting TCD Operating Parameters

The TCD can operate in constant temperature (CT) and constant voltage (CV) modes. The mode you choose depends on the concentration range of the sample and the required sensitivity. The CT configuration ensures the maximum linearity of the detector up to concentrations of 1% (g or mL). The CV mode extends the linearity range to higher values, but with a negative impact on sensitivity. After selecting the mode, you must program the following parameters:

- detector temperature
- filament temperature/filament voltage

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 GC column oven during the analysis.

The temperature/voltage applied to the filaments depends on the mode and the carrier gas used.

WARNING! In case of TCD with the polyimmide coated filaments, the maximum operating temperature is 300 °C for the TCD cell and 320 °C for the filaments.

Selecting an Operating Mode for High Thermal Conductivity Gases

When using hydrogen or helium, the operating mode you select depends on the type and concentration range of the compounds you are analyzing.

Using the Constant Temperature Mode

For samples in concentrations not exceeding 10% (g or mL), use the following values:

- detector temperature: higher than the maximum column oven temperature during the analysis
- filament temperature: 80–100 ºC above the detector temperature

This temperature difference results in a high sensitivity required for trace analysis (ppm). It also ensures a longer filament lifetime. Since the temperature remains constant, this mode considerably increases the filament life compared to other operating modes.

Using the Constant Voltage Mode

For samples in concentrations of a wide percentage range 1-100%; g or mL), use the following values:

- detector temperature: higher than the maximum column oven temperature during the analysis
- filament voltage: 5–7 V

In this operating mode, the detector response is linear up to the maximum concentrations.

Table 21-2 contains the selectable values for the detector temperature and the concentration range when using helium as the carrier gas.

Table 21-2. Selectable TCD Parameters

Concentration Range	Detector Temperature	Filament Temperature	Filament Voltage	Mode
ppm—5%	100 ºC	180 ºC	—	CT
0.5–100%	100 ºC	—	5 V	CV
ppm—5%	180 ºC	270 ºC	—	CT
0.5–100%	180 ºC	—	6 V	CV
ppm—5%	240 ºC	330 ºC	—	CT
0.5–100%	240 ºC	—	6 V	CV

When analyzing samples with a complete range of concentrations (ppm-100%), you can use different operating modes for different applications. The range between 5000 ppm and 5% allows a good linearity of the signal to linearize a series of data and obtain only one reading scale.

Selecting an Operating Mode for Low Thermal Conductivity Gases

When using nitrogen or argon, the operating mode you select depends on the type and concentration range of the compounds you are analyzing.

Using the Constant Temperature Mode

For samples in concentrations not exceeding 1% (g or mL), use the following values:

- detector temperature: higher than the maximum temperature reached by the column oven during the analysis, but not higher than 280–300 ºC
- filament temperature: 120–150 ºC above the detector temperature

Using the Constant Voltage Mode

When using low thermal conductivity gases, the temperatures reached by the filaments are very high for the low voltage supply. Table 21-3 contains the experimental filament temperature values corresponding to the applied voltages when using argon.

Table 21-3. Filament Temperature Values for Argon

Detector Temperature 100 ºC	Values					
Voltage (V)	5	6	7	8	9	10
Filament Temperature (ºC)	235	275	315	355	395	435

For samples with a wide range of concentration percentage (1-100%; g or mL), use the following values:

- detector temperature: higher than the maximum temperature reached by the column oven during the analysis, but not higher than 280–300 ºC
- filament voltage: 5 V

These temperature differences provide good sensitivity without compromising the filament lifetime.

TCD Menu

Table 21-4 shows the TCD control parameters.

Press LEFT DETECT or RIGHT DETECT to open the **`DETECTOR (TCD)`** menu, depending on the location of your detector.

Table 21-4. The Detector (TCD) Menu

Menu	Range	Comments
`RIGHT DETECTOR (TCD)`		This line is the menu title bar.
`Filament power`	On/Off	Press ON to turn on the filament power. Press OFF to turn off the filament.
`Fil status`	Ready/ Not Ready	This indicates the filament **Ready** or **Not Ready** status.
`Block temp`	On/Off, 50–450 ºC in 1 ºC increments	This is the detector temperature. Press ON to turn on the heater and display the actual and setpoint values. Press OFF to turn off the heater.
`Transf temp`	On/Off, 50–450 ºC in 10 ºC increments	This is the transfer line temperature for the heated zone between the oven and the detector cell. Press ON to turn on the heater and display the actual and setpoint values. Press OFF to turn off the heater. A value higher than or equal to the oven temperature, but lower than the detector temperature must be set.
`Const fil temp?`	Yes/No	Press YES to activate the constant filament temperature mode and display the `Fil temp (CT)` parameter. Press NO to display the filament voltage and maximum filament temperature parameters. The current operating mode (`CT`, `CV`, or `CC`) is displayed in parentheses.
`Fil temp (CT)`[1]	On/Off, 50–450 ºC in 10 ºC increments	This parameter indicates the filament temperature.

Table 21-4. The Detector (TCD) Menu (Continued)

Menu	Range	Comments
`Fil volts (CV)`[2]	5–15 V in 1 V increments	This parameter indicates the filament voltage.
`Fil temp limit`[2-3]	50–450 ºC	This parameter indicates the maximum filament temperature.
`Ref flow`	On/Off, 0–100 mL/min	This parameter indicates the reference gas flow. Press ON to turn on the flow and display the actual and setpoint values. Press OFF or 0 to turn off the flow.
`Mkup flow`	On/Off, 5–100 mL/min	This parameter indicates the make-up gas flow. Press ON to turn on the gas flow and display the actual and setpoint values. Press OFF to turn off the make-up flow.
`Carrier source`	R, L	When the GC has two injectors, this parameter tells the GC which inlet, left or right, is connected to the TCD. This parameter is used to protect the filaments on DGFC systems when the carrier supply is inadvertently shut off, such as following a septum replacement.

1. This parameter appears only if the `Const fil temp?` parameter is set to `Yes`.
2. This line appears only if `Const fil temp?` is set to `No`.
3. When the TCD with the polyimide coated filaments is used, the max temperature is 320 °C.

OPERATING SEQUENCE

Programming a TCD

Before you begin, do the following:

- Verify that all detector gases are connected, a column is correctly installed, and the system is leak free.
- Check the oven temperature and injector temperature.
- Check the carrier gas flow depending on the packed or capillary column in use.
- When two injectors are configured, scroll to `Carrier source` and specify the `Left` or `Right` channel from which the carrier gas is flowing.
- When a wide-bore or capillary column is used, make sure the make-up gas line is connected.

WARNING! Hydrogen is a potentially dangerous gas. Refer to *Using Hydrogen* on page xxviii for safety information.

1. Scroll to `Ref flow` and set the appropriate reference gas flow. If this value is `Off`, the filament power is disabled.
2. When make-up gas is required, scroll to `Mkup flow` and set the appropriate make-up gas flow rate.
3. Scroll to `Block temp` to enter the detector temperature.
4. Scroll to `Transfer temp` and set this temperature to a value higher or equal to the column oven temperature.
5. Scroll to `Const fil temp?` to select the operating mode. When constant filament temperature is required, press YES. Otherwise, press NO.
    - If `Y` has been entered, scroll to `Fil temp` and set the filament temperature. This value must always be higher than the detector temperature. The greater the difference between the two temperatures (ΔT), the higher is the detector sensitivity.

Set this value depending on the high or low thermal conductivity of the carrier gas in use.

- If `N` has been entered, scroll to `Fil volt` and set the filament voltage.
- Scroll to `Fil temp limit` and set the maximum filament temperature to protect the system. This value must always be higher than the detector temperature.

6. Scroll to `Filament power` and press ON. After a few seconds, the `Fil status` line displays a `Ready` message.
7. Scroll to `Carrier source` and press ENTER.
8. Scroll to the inlet connected to the TCD, `R` or `L`, and press ENTER.

If the reference gas or carrier gas is missing, the filament power turns off or will not switch on. The carrier source you select in step 8 indicates the source of the carrier gas for this filament protection sequence.

9. Press LEFT SIGNAL or RIGHT SIGNAL to open the detector **`SIGNAL (TCD)`** menu and verify the output signal.

Refer to the *Setting the TCD Signal Parameters* operating sequence on page 433 for instructions on setting the signal parameters.

OPERATING SEQUENCE

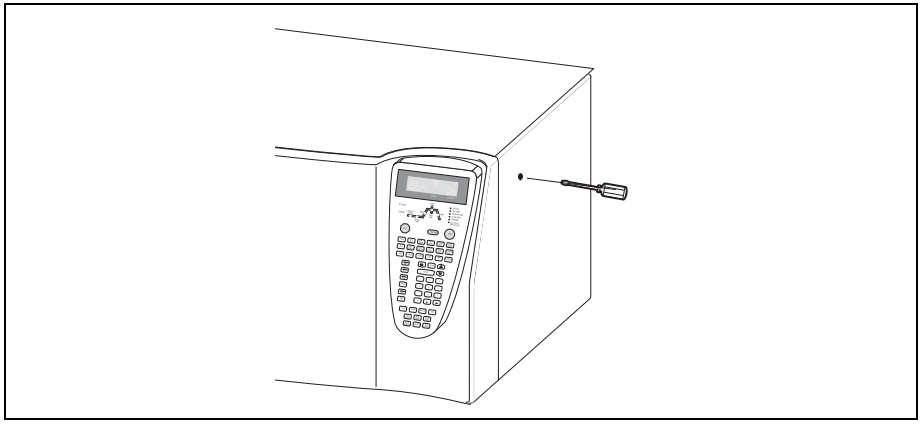
Setting the TCD Signal Parameters

1. Press LEFT SIGNAL or RIGHT SIGNAL to enter the detector **`SIGNAL (TCD)`** menu.
2. Scroll to `Gain (x1 or x10)` and set the desired value. When the gain is x10, the system sensitivity is higher. This amplifies not only the detector output signal, but also the electrical and mechanical noise.

3. If required, scroll to `Neg polarity` and press YES to reverse the polarity output signal as a function of the thermal conductivity of the carrier gas versus the sample.

4. With all gas flows and temperatures adjusted and stable, and with the filaments on and stable, scroll to `Offset` and press OFF.

5. Zeroing the Signal.
   Every time the set condition of filaments temperature and voltage are changed, an adjustment of the Zero level may be necessary in order to balance the bridge.Usually this operation is performed by scrolling to `Autozero` function and pressing ON. In case of a particularly unbalanced bridge, the Autozero function may not work. In that case it is necessary to adjust the coarse zero potentiometer of the detector control board by means of a little screwdriver until a signal of `1000` is visualized.



6. Turn `Baseline comp` ON if you want to compensate the baseline.

OPERATING SEQUENCE

Shutting Down the TCD

At the end of the analytical cycle, the filaments should be turned off and the carrier gas flow should be reduced to 50% of the normal operating flow to conserve gas supplies.

22

Pulsed Discharge Detector (PDD)

This chapter describes the operating sequences and principles for the Pulsed Discharge Detector (PPD).

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PDD Gas Supply 438
PDD Installation 443
PDD Menu 448

Operating Sequences

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Purging the Helium Purifier 441
Connecting the Gas Lines 442
PDD Cell Leak Check 445
System Leak Check 446
Programming a PDD 449
Setting the PDD Signal Parameters 450

PDD Overview

The Pulsed Discharge Detector (PDD), shown in Figure 22-1, is an universal and highly sensitive non-radioactive and non-destructive detector. It is based on the principle of the photoionization by radiation arising from the transition of diatomic helium to the dissociative ground state.

NOTE This detector does not use radioactive sources.

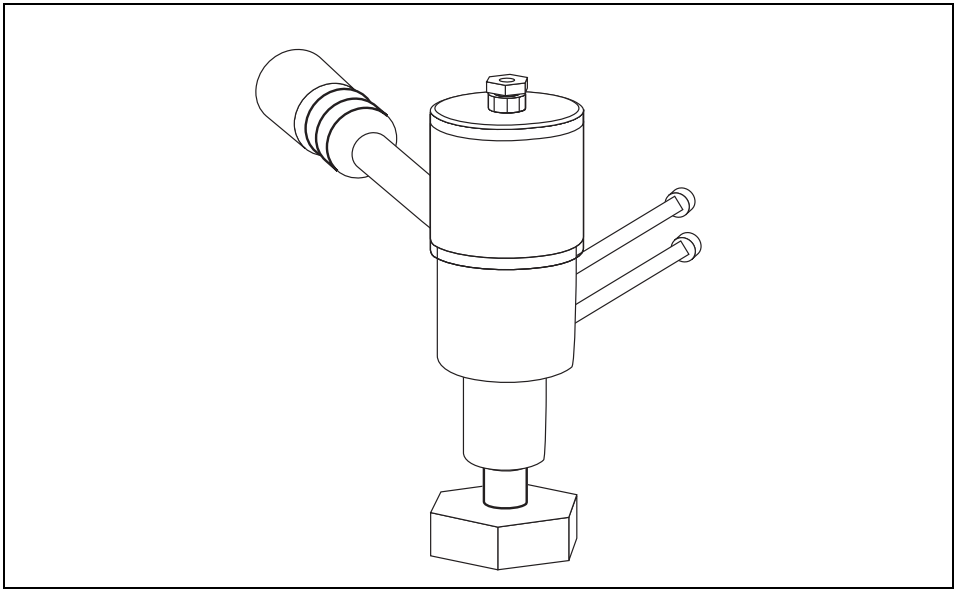
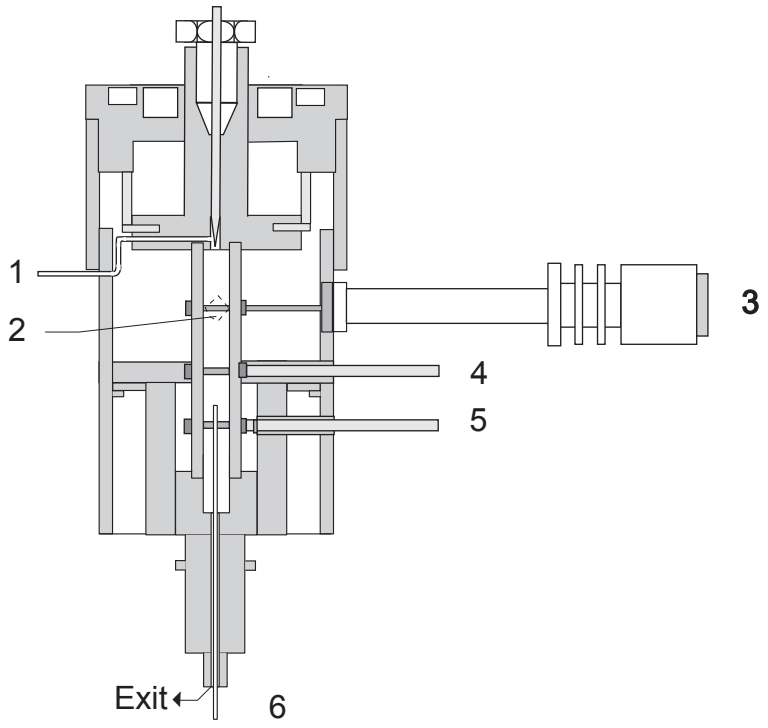


Figure 22-1. The Pulsed Discharge Detector

The response to organic compounds is linear over five orders of magnitude with minimum detectable quantities in the low picogram range. The response to fixed gases is positive with minimum detectable quantities in the low ppb 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.

PDD Principle

PDD detector consists of a quartz cell supplied from the top with ultrapure helium as discharge gas that reaches the discharge zone consisting of a couple of electrodes connected to a high voltage pulses generator (Pulsed Discharge Module)



1. Discharge gas (Helium) Inlet	2. Discharge Zone
3. High Voltage Discharge Electrode	4. Polarizing Electrode (Bias)
5. Collector Electrode (Signal)	6. Column

Figure 22-2. PDD (Cutaway View)

The eluants 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 eluants 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 outlet on the back of the instrument.

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.

PDD Gas Supply

PDD requires one gas flow only.

- discharge gas

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 60 psi (413 kPa).

Gas Purity

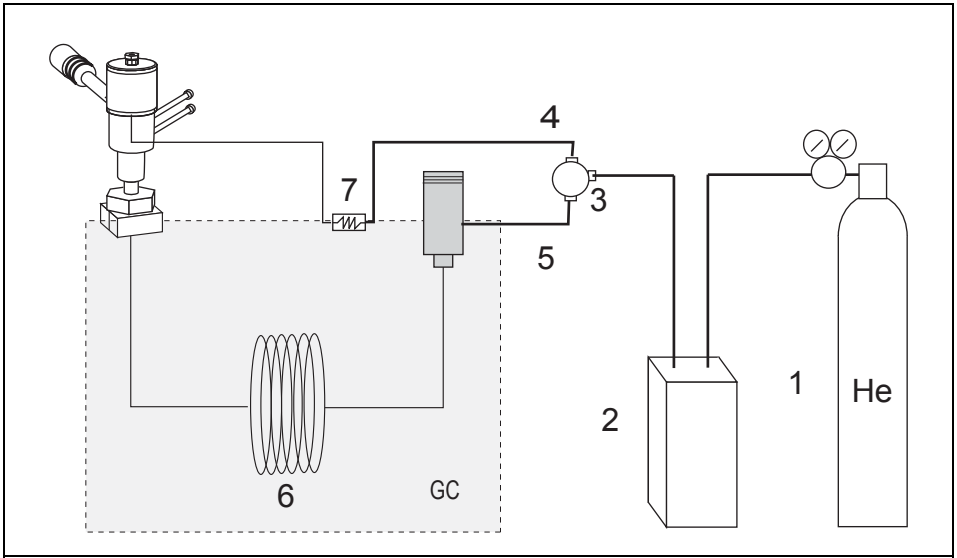
Helium must have a minimum purity of 99.999%, with < 20 ppm Ne impurity.

For trace analysis of fixed gases, it is strongly recommended 99.9999% purity helium with < 0.5 ppm Ne.

WARNING! The discharge and the carrier gases must always flow through the helium purifier.

Gas Lines Connections

Figure 22-3 shows the gas connections detector system diagram.



1. Helium Supply
2. Helium Purifier
3. 3-way connector
4. Discharge Gas Line
5. Carrier Gas Line
6. Column

Figure 22-3. Gas Connections

Before connecting gas lines verify that:

- The pressure regulators are commercial ultra-pure grade regulators with stainless steel diaphragms.
- The connecting tubes are thoroughly cleaned and baked before use.
- The gas regulator and the helium purifier must be properly purged. Refer to the following operating sequences for further details.

OPERATING SEQUENCE

Purging the Gas Regulator

1. Make sure that the on/off valve on the helium cylinder is completely closed.
2. Screw the fitting nut of the regulator into the helium cylinder. Go beyond finger-tight, but do not tighten the nut all the way because some leakage is required for purging operation.
3. Turn the output pressure regulating knob completely counterclockwise.
4. Open the cylinder on/off valve slightly and quickly close it again.
5. Adjust the tightness of the regulator connecting nut to allow a pressure reduction of about 690 kPa/sec (100 psi/sec).
6. When the pressure drops into the 1.4 - 3.4 MPa (200 - 500 psi) range, open the cylinder on/off valve slightly and quickly close it again.
7. Repeat the step 6 until it is certain that all the air is purged.
   On the final purge, tighten the regulator connecting nut as the pressure approaches the 2.1 - 3.4 MPa (300 - 500 psi) range.
8. Open the cylinder valve to pressurize the regulator once again.
9. Close the valve and observe the needle of the high pressure gauge for 15 minutes. If it does not move, there is no critical leak on the high pressure side of the regulator.

WARNING! Never use leak detecting fluids on any part of the system.

OPERATING SEQUENCE

Purging the Helium Purifier

1. Connect the helium cylinder pressure regulator to the inlet port of the helium purifier by using the appropriate connecting tube and fittings.
2. Turn the output pressure regulating knob clockwise until the gauge registers 345 kPa (50 psi)
3. Wait five minutes for equilibrium, then turn the regulating knob all the way counterclockwise.
4. Observe the needle of the output pressure gauge for 15 minutes. There will be a slight initial drop. If it does not move after that, consider all the connections are tight.
5. If necessary, use an electronic leak detector to locate any leaks. If a leak detector is not available, tighten all the fitting (including the output pressure gauge), and repressurize the system for another test.

WARNING! Never use leak detecting fluids on any part of the system.

6. Uncap the outlet tube of the helium purifier and purge the system for 15 to 30 minutes at 60 - 80 ml/min to eliminate air from the purifier getting material.

OPERATING SEQUENCE

Connecting the Gas Lines

1. Connect the helium purifier outlet port to a port of the 3-way connector provided by using the 1/16” OD connecting tube provided.

2. Connect the second port of the 3-way connector to the discharge gas inlet (calibrated restrictor), located on the rear panel of the GC, by using a sufficient piece of the 2x1 mm steeling steel connecting tube provided and the appropriate fitting.

3. Connect the last port of the 3-way connector to the DCC carrier gas inlet port, located on the rear panel of the GC, by using a sufficient piece of the 2x1 mm steeling steel connecting tube provided and the appropriate fitting.

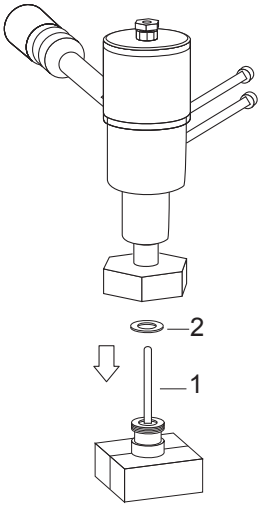
PDD Installation

This operation allows the correct installation of the PDD on your TRACE GC Ultra.

Material needed

- PDD fixing tool

CAUTION When packed columns are used (Only 1/8-inch OD), before installing PDD, verify that all the preliminary operations have been performed as described in *Connecting a Metal Packed Column to an PDD* operating sequence in Chapter 14.



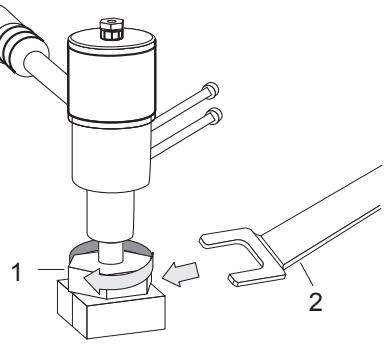
1. Fused Silica Capillary Tube or Capillary Column
2. Silver Ring

Figure 22-4. Installation of the PDD (1)

1. Place the PDD on the detector base body, paying attention to interpose the silver ring provided.

CAUTION Place carefully the PDD perfectly vertical paying attention to not damage the fused silica capillary tube or the capillary column.

2. Tighten the fixing nut by using the PDD fixing tool.

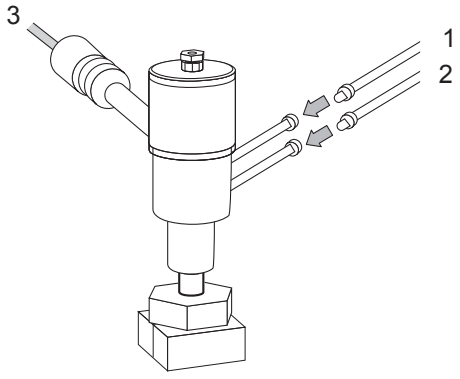


1. Detector Base Body 2. PDD Fixing Tool

Figure 22-5. Installation of the PDD (2)

3. Carefully connect the collector (signal) and polarizing (bias) cables coming from the detector control card to the detector cell.

4. Verify that the high voltage cable is properly connected to the pulsed discharge module.



1. Polarizing (Bias) Cable
2. Collector (Signal) Cable
3. High Voltage Cable

Figure 22-6. PDD Connecting Cables

Leak Check

It is critical for the system to be leak-tight. Leak test is strongly recommended before operating with PDD. Refer to the following operating sequences.

OPERATING SEQUENCE

PDD Cell Leak Check

Material required

- flowmeter
- sealing tool

WARNING! Do not use leak detecting liquids.

1. Open the discharge gas supply (helium).

2. Set an helium inlet pressure at 415 kPa (60 psi) to have a gas flow of 30 ml/min.

You may measure the helium discharge flow rate at the exit of the pneumatic module on the rear of the GC.

3. Cap the discharge gas exit on the rear of the GC by using the sealing tool provided.
4. Disconnect the outlet column end from the detector base body.
5. Plug the column connection of the detector base body.
6. Monitor the pressure by using an external gauge (e.g. the gauge installed on the bottle).
7. Let the system pressurize, then turn off the discharge gas flow. The shown values should not change. If the values drop down, one or more leaks are present. In this case:
8. Check the accessible, critical connections with a handheld electronic leak detector to find possible leaks.
9. If no leak is detectable in this way, contact your customer support organization. Refer to Appendix B, *Customer Communication*, for contact information.

OPERATING SEQUENCE

System Leak Check

With the PDD installed and the column properly connected, operate as follows:

1. Open the carrier and the discharge gas supply (helium).

2. Set the helium discharge pressure at 415 kPa (60 psi) to have a gas flow of 30 ml/min.

NOTE You may measure the helium discharge flow rate at the exit of the pneumatic module on the rear of the GC.

1. Cap the discharge gas exit on the rear of the GC by using the sealing tool provided.
2. Turn off the split and septum purge vents (if any).
3. Set the injector inlet to 100 kPa.
4. Wait until the system is equilibrated.
5. Turn off the inlet pressure and the discharge gas pressure.
6. The shown values should not change. If the values drop down, one or more leaks are present. In this case:
7. Check the accessible, critical connections (column to injector, column to detector, split and purge valves, septum cap) with a handheld electronic leak detector to find possible leaks.
8. If no leak is detectable in this way, contact your customer support organization. Refer to Appendix B, *Customer Communication*, for contact information.

PDD Menu

The **DET(PDD)** menu contains the PDD control parameters.
Press LEFT DETECT or RIGHT DETECT to open the menu shown in Table 22-1.

Table 22-1. Detector (PDD) Menu

Menu	Range	Comment
RIGHT DET (PDD)		This line is the menu title bar.
Pulse generator	On/Off	This line indicates the pulsed discharge module status. Press ON to turn on the voltage supply from the PDD control card to the module which will generate the high voltage required to supply the detector. Press OFF to turn off the module.
Base temp	On/Off, 0–450 °C	This indicates the detector base body temperature. Press ON to turn on the heater and display the actual and setpoint values. Press OFF to turn off the heater and display the actual value.
Signal pA	Not editable	This parameter shows the standing current level in picoamperes.

OPERATING SEQUENCE

Programming a PDD

Before you begin, do the following:

- Verify that helium purifier and discharge gas are connected, a column is correctly installed, and the system is free of leaks.
- Check the oven temperature and injector temperature.

1. Press LEFT DETECT or RIGHT DETECT to open the **`DET(PDD)`** menu.
2. Scroll to `Base temp` and set the detector base body temperature according to the analytical requirement.
3. Scroll to `Pulse generator` and turn it ON.
4. Read the `Signal pA` value.
   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.

After a short time, the baseline should stabilize to the standing current level of the system.

5. Press LEFT SIGNAL or RIGHT SIGNAL to open the detector **`SIGNAL (PDD)`** menu and verify the output signal.

Refer to the *Setting the PDD Signal Parameters* operating sequence on page 450 for more information.

OPERATING SEQUENCE

Setting the PDD Signal Parameters

1. Press LEFT SIGNAL or RIGHT SIGNAL to enter the detector **`SIGNAL (PDD)`** menu.
2. Scroll to `Range 10^ (0...3)` and set the electrometer amplifier input range. `0` (10^0) is the most sensitive.
3. Turn `Analog filter` ON if you want to filter the output signal.
4. Scroll to `Autozero` and press ON.
5. If offset is required, scroll to `Offset` and enter a numeric value or press ON to recall the last offset from memory.
6. Turn `Baseline comp` ON if you want to compensate the baseline.

NOTE

If the `Range 10^` is set 2 or 3, the small variation of the output signal is not detected. For this reason, the, `Signal pA`, parameter will be not displayed in the **`DETECTOR PDD`** menu.

SECTION

VI

Autosamplers

This section contains informations about AI 3000/AS 3000 programming with the TRACE GC Ultra keypad.

Chapter 23, *AI 3000 / AS 3000 Autosampler*, describes how to program and control the AI 3000 / AS 3000 autosampler by using the TRACE GC Ultra keypad.

23

AI 3000 / AS 3000 Autosampler

This chapter describes how to program and control the AI 3000 / AS 3000 autosampler by using the TRACE GC Ultra keypad.

Chapter at a Glance…

Autosampler Overview 453
AI 3000 / AS 3000 Autosampler Menu 456

Autosampler Overview

This paragraph contains the instructions to program AI 3000 / AS 3000 parameters.

The autosampler AI 3000/AS 3000 functions can be controlled from:

- a Thermo Scientific Data System referring to the instructions reported in the relevant operating manual.
- TRACE GC Ultra keypad, referring to the instructions reported in this chapter.

The functions that the TRACE GC Ultra can control include:

- Injection prewash volume and solvent
- rinse cycles, volume, and solvent
- sample volume

- injection, including special instructions such as:
  - number of plunger strokes
  - viscosity delay
  - air gap volume and mode
  - injection speed
  - pre- and post-injection delay time
- postwash cycles and solvent

Groups of samples may be automatically run under different analytical conditions programming a *sequence* of samples. A *sequence* describes how samples are treated in the injection stage. The sequence includes the instructions for sampling, number of samples and their position on the sample tray. Beside the sequence specifies the method that will be used to process each samples group.
Refer to paragraph *Sequence Programming* on page 489 for instructions.

NOTE All autosampler functions can be programmed into an analytical method. Refer to Chapter 26, *Using Analytical Methods*, for more information on developing a method. A sequence cannot be programmed into a method.

Compatible Hardware

Several autosampler models can work with the TRACE GC Ultra. The menus and instructions in this chapter apply to the AI 3000 / AS 3000.

Setting Up the Autosampler

When the GC is switched on, the presence of the autoinjector/sampler and its configuration is automatically acknowledged.

The type of configuration is displayed by pressing CONFIG and selecting **AUTOSAMPLER**.

The following non editable message is displayed according to the autoinjector/sampler installed:

```
     CONFIG AUTOSAMPLER

 Sample Tray            105
```

for the AS 3000.

```
     CONFIG AUTOSAMPLER

 Sample Tray              8
```

for the AI 3000.

NOTE Should the autoinjector/sampler not be installed or correctly connected, the TRACE GC Ultra will display the message NO AUTOSAMPLER INSTALLED.

AI 3000 / AS 3000 Autosampler Menu

To set the parameters of the autoinjector/sampler method, press AUTOSAMPLER to open the **AUTOSAMPLER** menu shown in table 23-1.

Table 23-1. Menu of the AI 3000/AS 3000

Menu	Range	Comments
AUTOSAMPLER		This line displays the menu title.
Sample volume	0–5 µL with increments of 0.1 µL	This parameter allows to set the sample quantity to be injected.
Sample rinses	0–15	This parameter allows to set the number of syringe pre-washings with the sample.
Plunger strokes	0–15	This parameter allows to set the number of plunger strokes to eliminate air bubbles forming during the sample withdrawal.
Pre wash solvent	A, B, C, D, A+B, C+D	This parameter allows to select the vial, or combination of two vials, containing the washing solvent. Press MODE/TYPE to select the solvent vial to be used.
Pre wash cycles	0–15	This parameter allows to set the number of syringe pre-washings with the selected solvent.
Post wash solvent	A, B, C, D, A+B, C+D	This parameter allows to select the vial, or combination of two vials, with the washing solvent. Press MODE/TYPE to select the vial with the solvent to be used.
Post wash cycles	0–15	This parameter specifies the number of syringe post-washings with the solvent selected.
Extended control	See table 23-2	This parameter allows to set optional injection parameters. Press MODE/TYPE to enter the submenu.
When no vial abort	See table 23-3	This line shows the menu title.

Extended Control Menu

In this menu it is possible to set how long the syringe needle must remain inside the injector without injecting. This allows to avoid discriminations caused by evaporation of the sample contained in the syringe needle. In this way the sample is completely drawn into the syringe and the needle left inside the injector and heated for a few seconds before injecting.

Table 23-2. Extended Control Menu

Menu	Range	Comments
`EXTENDED CONTROL`		This line shows the menu title.
`Viscous sample`	Yes, No	This parameter defines the speed at which the sample is drawn from the vial as a function of the sample viscosity. Select NO (default value) if the sample has low viscosity. Select YES if the sample has high viscosity.
`Sampl. depth`	Bottom, Center selectable in the submenu	This parameter determines the penetration depth of the syringe needle into the vial. Press MODE/TYPE or ENTER to enter the submenu. Selecting `Bottom` (default value) the needle goes down to the vial bottom. Selecting `Center` the needle goes down to half vial.
`Inj. Depth`	Standard, Minimum selectable in the submenu	This parameter determines the penetration depth of the syringe needle into the injector. Press MODE/TYPE or ENTER to enter the submenu. Selecting `Standard` (default value) the needle goes down to the maximum depth allowed. Selecting `Minimum` the needle enters the injector and stops immediately beyond the septum (*Cold Needle Technique*).
`Pre dwell time`	0–63 sec	This parameter specifies how long the syringe needle remains inside the injector without injecting (*Hot Needle Technique*).

Table 23-2. Extended Control Menu (Continued)

Menu	Range	Comments
Post dwell time	0–63 sec	This parameter specifies how long the syringe needle remains inside the injector after injection.

When No Vial Abort Menu

Table 23-3. When No Vial Abort Menu

Menu	Range	Comments
ON MISSING AS VIAL		This line shows the menu title.
Skip to next		If this function is selected, the autoinjector/sampler skips a missing vial and goes to the next one. The sample sequence and the sample table are not affected.
Abort sequence		If this function is selected, the sequence is aborted after a missing vial.

SECTION

VII

Automation and Manual Control

This section contains descriptions of automated and manual control options and sequences for the TRACE GC Ultra.

Chapter 24, *Automated Functions*, shows you how to automate signal, valves, and external events by scheduling them either in real time (clock table events) or at certain points during a run (run table events). It also discusses the run log, an automated record of run deviations.

Chapter 25, *Manual Functions*, describes how to control signal and valve events manually.

24

Automated Functions

This chapter shows you how to automate signal, valves, and external events by scheduling them either in real time (clock table events) or at certain points during a run (run table events). It also discusses the run log, an automated record of run deviations.

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Programming External Event Default Conditions 471
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The Clock Table

Clock table events happen at certain times on specific days, based on a real-time clock. The real-time clock, once set, is backed up by a battery that maintains the clock time even when the GC is powered down. Among the functions you can program are:

- loading a method
- starting the GC
- starting a sequence
- opening or closing valves
- starting external events for other devices, such as a mass spectrometer or automatic actuated valves

The devices you can control depend on the options you purchased and how your TRACE GC Ultra was configured at the factory.

If no events are programmed, the **CLOCK EVENTS** menu looks like the one on the left in Figure 24-1. The menu on the right shows a **CLOCK EVENTS** menu with eight events. You can store up to 10 events.

In Figure 24-1, the right **CLOCK EVENTS** menu specifies several events.

The TRACE GC Ultra will load Method #10 at 4:00 A.M. At 4:56 A.M., `Valve 2` shuts off. Then `External Event #1` turns on and `Valve #1` (a gas sampling valve) loads one minute later. `External Event #1` turns off at 5:00 A.M. `Sequence #10` begins running at 7:00 A.M. (using `Method #10` loaded earlier).

The events shown in the right-hand menu in Figure 24-1 will occur every day because the `Mode` parameter is set to `cont cycle` (continuous cycle). You can schedule events to happen:

- once
  Use `Single cycle` to schedule a one-time event.
- every day
  Use `Continuous cycle` to prepare and start the TRACE GC Ultra for each day's first run.

- on certain days
  Use `Specific cycle` to specify an event to happen on specific days of the week.

You can also discontinue clock time events for a time.

```
   CLOCK EVENTS
<none>
Add clock event <
Mode: not active
```

```
        CLOCK EVENTS                ↓
04:00      Load meth          10 <
04:56      Valve 2            Off
04:57      External Event 1   On
04:58      Valve 1            Load
04:59      Valve 1            Inj
05:00      External Event 1   Off
07:00      Start seq          10
Add clock event
Mode:      Cont cycle
```

Figure 24-1. Two Clock Events Menus (Empty and Loaded)

NOTE

When clock events fall during a run or an active sequence, the TRACE GC Ultra ignores them. For instance, if you have scheduled a bakeout at 9:00 A.M., it will not occur if the TRACE GC Ultra is running a sequence of samples. To program an event to occur during a run, refer to *The Run Table* on page 467 for more information on run time events. You can include run table events in an analytical method, but not clock table events.

OPERATING SEQUENCE

Creating a Clock Time Event

Use the following sequence to enter new clock time events.

1. Press the CLOCK TABLE key.

2. Scroll to `Add clock event`.

3. Press ENTER or MODE/TYPE to display the **`SELECT EVENT TO ADD`** submenu, shown in the first column of Table 24-1.

4. Scroll to the type of event you want to add. Press ENTER or MODE/TYPE to display the submenu for that item.

Table 24-1. Select Event to Add Menu and Submenus

Menu	Submenu	Comments
`SELECT EVENT TO ADD`		This line is the menu title bar.

`Load Method <`	`LOAD METHOD`
	`Method no.`
	`Clock time`
		With the numeric keypad, enter a method number (1–10) and a time in hours and minutes (00:00–23:59).

`Start GC`	`CLOCK TIME EVENT`
	`Start GC at`
	`Clock time`
		With the numeric keypad, enter a time in hours and minutes (00:00–23:59).

`Start seq`	`CLOCK TIME EVENT`
	`Start sequence`
	`Sequence no.`
	`Clock time`
		With the numeric keypad, enter a sequence number (1–5) and a time in hours and minutes (00:00–23:59).

`External Event`	`SELECT EXTERNAL EVENT to add`
	`External Event #1`
	`----------`
	`External event #8`
	`Clock time`
	`Setpoint.`
		Press ENTER to enter submenu
		With the numeric keypad, enter an event number and a time in hours and minutes (00:00–23:59).

`Baseline comp`	`CLOCK TIME EVENT` `Start baseline compensation at Clock time`	With the numeric keypad, enter a time in hours and minutes (00:00–23:59)

5. If you are setting up a method or a sequence, enter a method number (1–10) or a sequence number (1–5) and press ENTER.

6. Use the numeric keypad to enter the time you want the event to take place, based on a 24-hour clock. You must enter four digits. For example, for 3:30 p.m., type `1530`. Press ENTER to record the time in memory.

7. If you are programming an external event, use the ON/YES or OFF/NO key to enter the setpoint. If you program `External Event #1` to turn on at a certain time, you should add another event to turn it off at a later time.

NOTE

Because the TRACE GC Ultra ignores any clock table event that falls during a run, you should program any event you want to occur during a run in the run table. Refer to *The Run Table* on page 467 for more information about run time events. You can include run table events in an analytical method, but not clock table events.

8. Press CLEAR twice to return to the main **`CLOCK EVENTS`** menu.

OPERATING SEQUENCE

Programming Occasionally Occurring Events

The `Mode` function lets you set the clock time events to occur at different times of the week. Use the following sequence to program the days the events will happen.

1. Scroll through the main **`CLOCK TABLE`** menu to `Mode`. Press ENTER or MODE/TYPE to display the **`CLOCK EVENT MODE`** submenu, shown in Table 24-2.

Table 24-2. Mode Submenus

Menu	Submenus	Comments
`CLOCK EVENT MODE`		This line is the menu title bar.
`Not active`		Choose this option to suspend the clock events indefinitely.
`Single cycle`		Choose this option to make the clock events occur only once.
`Continuous cycle`		Choose this option to make the clock events happen every day.

Table 24-2. Mode Submenus (Continued)

Menu	Submenus	Comments

`Specific cycle`	`CLOCK EVENT MODE`
	`Use ENTER to selct/ deselct active days`
	`CLEAR to exit`
	`Sunday`
	`Monday`
	`Tuesday`
	`Wednesday`
	`Thursday`
	`Friday`
	`Saturday`
		Select `Specific cycle` and press ENTER or MODE/TYPE to display the submenu. Select the days you want the clock events to occur and press ENTER after each selection. The asterisk will appear on chosen days.

2. Select one of the choices in the **`CLOCK EVENT MODE`** submenu, depending on how often you want the event to occur. Press ENTER. If you want the event to occur on specific days, select `Specific cycle` and press ENTER or MODE/TYPE.

3. In the `Specific cycle` submenu, select the day you want the events to occur and press ENTER. Repeat this step to schedule additional days.

4. Press CLEAR twice to return to the **`CLOCK EVENTS`** main menu.

OPERATING SEQUENCE

Editing a Clock Time Event

You can change the time of day that a clock time event occurs. However, if you want to change the type of event that occurs, you must delete the current event and add a new one. For example, if you want External Event #2 to turn on at 5:00 A.M. instead of External Event #1, delete the event `05:00 External Event#1 On` using the *Deleting a Clock Time Event* operating sequence. Use the *Creating a Clock Time Event* operating sequence to add a new event for Valve 2 to load at 5:00 A.M.

Use the following sequence to edit the time a clock event occurs.

1. Press CLOCK TABLE and scroll to the item you want to edit.
2. Press ENTER or MODE/TYPE to display the submenu for that item.
3. Using the numeric keypad, enter a new time for the event. Press ENTER. The **CLOCK EVENT** menu now shows the item at its new time.
4. Press CLEAR twice to return to the **CLOCK EVENTS** main menu.

OPERATING SEQUENCE

Deleting a Clock Time Event

Use the following sequence to delete an event from the **CLOCK EVENT** menu.

1. Press CLOCK TABLE and scroll to the item you want to delete.
2. Press CLEAR once.
3. The following message appears on the display:

   ```
   You are about to delete the above entry. Delete it? Y/N
   ```

   Press YES to delete or NO to keep the event.

The Run Table

You can program events to happen during a run. For instance, a valve could open two minutes into a run. You can include a run table for each analytical method you create. You can program:

- an output signal adjustment, such as auto zero see *Controlling Output Signals* in Chapter 25 for a discussion of signal compensation)
- a valve to open or close (see *Controlling Output Signals* in Chapter 25 for a discussion of valve types and options)

- an external event from another device

NOTE

Eight external events are available, but each extra valve (those other than inlet valves) takes up two external events: one to open the valve, the other to close it. If you have one valve configured, only six external events will appear on your menus.

Whereas the clock table events occur on a 24-hour real-time clock, the Run Table events occur on a decimal-minute clock that begins counting when the run starts.

Figure 24-2 shows two **RUN TIME EVENTS** menus, one without entries and one with several entries.

```
    RUN TIME EVENTS
<none>
Add run time event<
Ext. event defaults
```

```
   RUN TIME EVENTS          ↓
 00.00  RFID          Off <
  1.00  RFID            On
  1.00  RFID       range 0
 17.00  RFID       range 1
 35.00  RFID       range 0
Add run time event
```

Figure 24-2. Two Run Time Events Menus (Empty and Loaded)

The first three events shown in the loaded menu in Figure 24-2 concern the right detector, a flame ionization type (RFID). It starts the run in the off position. At three minutes it turns on and adjusts its signal to the highest sensitivity.

At 17 minutes the RFID again adjusts the range, this time to the lowest sensitivity. At 35 minutes it returns the range to 0.

You can program events like those shown in Figure 24-2 with the **Run Time** menu and submenus.

OPERATING SEQUENCE

Creating a Run Time Event

Use the following sequence to enter new run time events.

1. Press RUN TABLE and scroll through the menu until the selection arrow points to `Add run time event`.

2. Press ENTER or MODE/TYPE to display the first **`RUN TABLE`** submenu.

```
  SELECT EVENT to add

Signal                    <
External Event
```

3. Scroll to the type of event you want to add: `signal` or `external`. Press ENTER or MODE/TYPE to display the submenu for that item, shown in the first column of Table 24-3.

Table 24-3. Select Event to Add Options and Submenus (1)

Option	Submenu 1	Comments
`Signal`	`Select Parameters to add`	This line is the submenu title bar.
	`RFID Autozero`	Choose this option to perform autozeroing
	`RFID Range (0..3)`	Choose this option to adjust detector Range

`External Event`	`Select Event to add`
	`External event #1`
	`----------`
	`External event #8`
		Choose this option to program up to eight external events

4. Select the appropriate kind of `signal` or `external` event and press ENTER or MODE/TYPE to open another submenu, shown in the first column of Table 24-4.

Table 24-4. Select Event to Add Options and Submenus (2)

Option	Submenu 1	Comments
RFID Autozero	RUN TIME EVENT RFID auto zero Run time	With the numeric keypad, enter a time (0.00–999:99).
RFID Range (0..3)	RUN TIME EVENT RFID Range Run time Range 10^	With the numeric keypad, enter Range number [(0 - 3), (0 - 2 for FPD)] and time (0.00–999.99).
External event #1 < ---------- External event #8	RUN TIME EVENT External event #1 Run time Setpoint	With the numeric keypad, enter an a time (0.00-999.99) and the On/Off setpoint

For example, if you previously selected a valve event, at this stage you designate which valve will be affected. If you choose Switching Valve #1, pressing ENTER will bring up the **RUN TIME EVENT VALVE #1 SWITCHING** menu.

NOTE If you wish to set a programmed external event to be the default condition for an external device, refer to the *Programming External Event Default Conditions* operating sequence on page 471.

5. Fill the parameter fields by using the numeric keypad or the ON/YES and OFF/NO keys. Parameters will differ among submenus, but each will require a run time in addition to its other settings.

6. Use the numeric keypad to enter an amount of time after the run starts for the event to take place. The run start time is 00.00. For example, for three minutes into the run, type `3` or `3.00`. Press ENTER to record the time in memory.

NOTE

Time units for run time events are displayed in hundredths of a minute, not minutes and seconds. For example, to program an event to occur 3 minutes and 30 seconds into a run, you would enter 3.5 rather than 3.30.

7. Press CLEAR three times to return to the **RUN TIME EVENTS** main menu.

OPERATING SEQUENCE

Programming External Event Default Conditions

Before you can perform this sequence, you must have programmed the external device event as described in the *Creating a Run Time Event* operating sequence on page 469.

1. Press RUN TABLE, scroll to `Ext. event defaults` and press ENTER.
2. Scroll to the external event you to set as the default condition:
   - Press ON to set the external event device default condition to `On`.
   - Press OFF to set the external event device default condition to `Off`.

The external device will return to the condition specified by the external event you have programmed to be the default whenever the GC is in **Standby** mode.

OPERATING SEQUENCE

Editing a Run Time Event

You can change the time a run time event occurs. However, if you want to change the type of event that occurs, you must delete the current event and add a new one.

For example, if you want the right FID detector to turn on at 1:00 A.M. instead of the left NPD, delete the event `1.00 LNPD On` using the *Deleting a Run Time Event* operating sequence on page 472. Using the *Creating a Run Time Event* operating sequence on page 469, add a new event that reads `1.00 RFID On`.

To edit the time of a run event, use the following sequence:

1. Press RUN TABLE and scroll to the item you want to edit.
2. Press ENTER or MODE/TYPE to display a submenu. Repeat Steps 1 and 2 until you reach the final submenu for the specific event, such as the **RUN TIME EVENT EXTERNAL EVENT #2** submenu.
3. Using the numeric keypad, enter a new time for the event. Press ENTER. The **RUN TIME EVENT** menu now displays the item at its new time.

OPERATING SEQUENCE

Deleting a Run Time Event

Use the following sequence to delete a run time event from the **RUN TIME EVENT** menu.

1. Press RUN TABLE and scroll through the menu to the item you want to delete.
2. Press CLEAR.
3. A message appears on the display:

4. `You are about to delete the above entry. Delete it? Y/N`

   Press YES to delete or NO to keep the event.

Run Log

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 RUN LOG. The Run Log is cleared and reset at the beginning of the next run.

25

Manual Functions

This chapter describes how to control signal and valve events manually.

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Setting the Valve Position 477

Controlling Output Signals

The TRACE GC Ultra provides output signals in two ways:

- digital data for a computerized data system
- 0–1 V and 0–10 V outputs for analog systems such as integrators

Each installed detector has a corresponding output signal; the left detector transmits the left signal.

To see a signal's current output, press either RIGHT SIGNAL or LEFT SIGNAL. The **SIGNAL** menu appears. Use the editable items to make the output more meaningful or measurable by:

- shifting (offset)
- amplifying to focus on certain peaks (range or gain)
- filtering electronic noise or drift (analog filter)
- forcing output values to start at zero (autozero)

Using these features can increase the accuracy of your analyses. Most can be set from the **SIGNAL** menu.

You may set these options at any time during a run. The changes you make in this menu during a run will override the run table's programmed instructions that have already occurred. However, subsequent run table instructions will override the earlier manual adjustments.

Table 25-1 describes each item of the **SIGNAL** menu. The menu will vary, depending on the detectors installed.

Table 25-1. Signal Menu

Menu	Range/Options	Comments
RIGHT SIGNAL (PID)		This line is the menu title bar.
Output	Not editable 0–1,100,000 unitless	This parameter displays a 20-bit digital output signal corrected by any items chosen from this menu.
Offset	On/Off, 0–65535	This parameter shifts the output signal to bring baseline within range.
Auto zero?	Yes/No	This parameter automatically adjusts the offset to zero (a digital signal of 1000).
Range=10^(0...3)[1]	0–2 FPD, 0–3 for all other detectors	This parameter attenuates the signal by powers of 10. Lower numbers are more sensitive.
Analog filter[1]	On/Off	This parameter reduces fast, spurious noise.
Baseline compensation	On/Off	This parameter allows baseline compensation function

1. Not displayed for TCD, PDD or ECD.

When To Use Signal Correction

If the chromatogram's baseline is too high…	Adjust the `Offset`.
If you want to automatically adjust the offset…	Use the `Autozero` feature.
If the peaks are saturating the detector…	Set the `Range` higher.
If the signal is too low to give a meaningful reading with an ionization detector...	Set the `Range` lower.
If you're seeing significant high frequency noise…	Turn on the `Analog filter`.

Controlling Valves

You can manually open or close valves before or during a run, overriding instructions from the run table. You can affect the inlet valves and up to eight external valves.

NOTE Each external valve uses one external event. You can have up to eight external events.

Types of Valves

Possible valve types for the TRACE GC Ultra and external devices are:

- septum purge
- split
- secondary cooling
- solvent vapor exit
- gas sampling
- switching
- stream select (multiposition)
- solvent
- Backflush

Most of these can be opened or closed using the ON/YES and OFF/NO keys from the **VALVES** menu. The exceptions are:

- gas sampling, which reads `Load` (press OFF/NO) or `Inject` (press ON/YES).
- `Stream select` (multiposition), which requires the port position of the valve.

Table 25-2 shows a sample **VALVES** menu and submenus.

Table 25-2. Valves Menu and Submenu

Menu	Submenu	Comments for Menu
VALVES		This line is the menu title bar.
Inlet valves	INLET VALVES R septum purge R split valve L SVE valve L sec cool valve	This parameter controls the valves for S/SL and PTV inlets only.
#1 Switching Switch valve default	SWITCHING VALVE	This valve switching parameter may be set to On or Off.
#2 Gas sample	SAMPLING VALVE	For the gas sampling valve parameter, the range is Inj=On, Load=Off
#3 Stream select		This parameter controls a multiposition valve

OPERATING SEQUENCE

Setting the Valve Position

Use the following sequence to manually set valve positions.

1. Press VALVE to open the **VALVES** menu.
2. Select either the inlet valves or one of the auxiliary valves.
   - For inlet valves, press ENTER or MODE/TYPE to move to the submenu. Select the appropriate position for the valve and press ENTER.

NOTE Inlet valves appear on the menu only if an S/SL or PTV inlet has been installed.

   - If the external valve is a multiposition valve, press ENTER or MODE/TYPE to move to the submenu. Enter the proper position number and press ENTER.
   - For all other kinds of external valves, select the appropriate position (on or off) for the valve and press ENTER.

   The appropriate action takes place immediately, overriding any programming.

SECTION

VIII

Methods and Sequences

This section contains information on programming analytical methods and using them in autosampler injection sequences.

Chapter 26, *Using Analytical Methods*, describes how to set up analytical methods that run automatically when specified.

Chapter 27, *AI 3000 / AS 3000 Autosampler Sequences*, contains the instructions to programming a sample sequence with the TRACE GC Ultra keypad when an AI 3000 / AS 3000 autosampler is used and how to set up ranges of samples to run automatically.

26

Using Analytical Methods

This chapter describes how to set up analytical methods that run automatically when specified.

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Method Parameters 482

Operating Sequence

Creating or Editing a Method 484
Storing a Method 487

Introduction

A *method* controls the function of the gas chromatograph during analytical runs. You may specify parameters for any zone and device (including temperature ramps in the oven menu), as well as run table timed events and autosampler parameters.
Up to ten methods may be programmed and stored in the TRACE GC Ultra in addition to the default method.

When an autosampler is used, you may associate different methods to run the analyses of group of samples connected to each other programming a sample *sequence*.

A sample *sequence* is basically a table where different batches of sample vials, accommodated in the autosampler sample tray, are linked together with different methods. Each step of the sequence requires the identification of each batch setting and the method to be used to analyze it.

See also Chapter 27, *AI 3000 / AS 3000 Autosampler Sequences*.

Method Parameters

You may specify many parameters in a method, such as:

- initial oven temperature
- up to seven temperature ramps
- post run oven temperature
- length of time to hold post run temperature
- initial and final carrier gas pressure
- detector types and parameters
- inlet types
- timed events in the run table
- autosampler parameters

When you press the **METHOD** key, the list of stored methods appears.

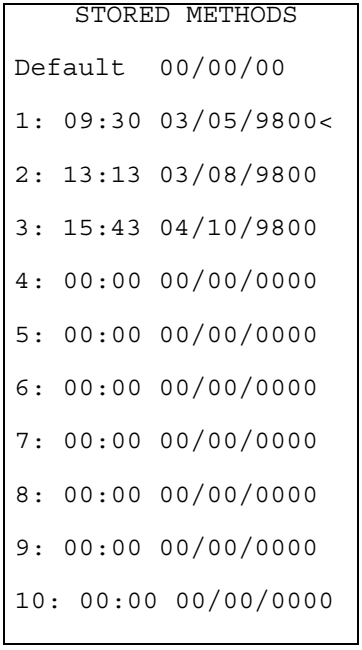

Figure 26-1. Stored Methods Menu

The methods are identified by a numeral. The menu shows the time and date they were created or modified. A row of zeros indicates that no method has been stored for that number.

You can store ten methods in the TRACE GC Ultra. A default method is programmed at the factory. If you are using a data system, you may store as many methods as your hard drive permits.

OPERATING SEQUENCE

Creating or Editing a Method

You can create a new method or edit a stored method during a run.
Press EDIT/ACTIVE to put the TRACE GC Ultra into the editing mode. Your edits do not affect the current run.

To create a method, press the METHOD key. Select a method number that shows zeros. Press ENTER, then select STORE from the menu shown in Figure 26-2.

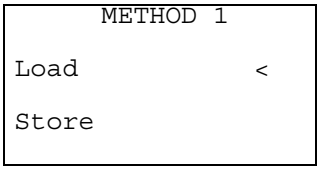

Figure 26-2. Method Menu

To edit a method, select a method number that shows a time and date and press ENTER. When editing is done, press STORE on the TRACE GC Ultra keypad.

NOTE

You can create or edit a method during a run by pressing EDIT/ACTIVE. The Non-Active LED will light. The parameters that appear are those last edited, not necessarily those currently running. (You may select a method from the stored list at this time.) When you have finished resetting the parameters and have stored them, press EDIT/ACTIVE again to return to the active mode. Your editing does not affect the current run.

The **OVEN** menu appears after you choose a method to edit or create. For more information on the **OVEN** menu, refer to Chapter 13, *The Column Oven*.

Initial Conditions

You need to set the initial oven temperature and the length of time the oven will remain at that temperature after the run begins.

1. In the **OVEN** menu, select Temp. Use the numeric keypad to type the temperature for the beginning of the run and press ENTER. The number on the right (the setpoint) will change to reflect your edit; the number on the left

shows the actual temperature of the oven. It will begin to change to meet your specifications.

2. Scroll to `Initial time`. Type the length of time in minutes that the oven should remain at the initial temperature after the run starts.

NOTE During a run, the Initial Temp LED will light when the TRACE GC Ultra receives the start signal and will remain lit during the initial time. In the example in Figure 26-3, the Initial Temp LED will stay on for two minutes.

OVEN		↓
Temp	40 40	<
Initial time	2.00	
Ramp 1	7.0	
Final temp 1	250	
Final time 1	10.0	
Ramp 2	Off	
Post run temp	300	
Post run time	9.50	
L Post pres	70	
R Post pres	70	

Figure 26-3. Sample Oven Menu

Ramps

You can specify up to seven temperature ramps in a method. After you have specified one ramp, the display presents options for the next ramp. Use the following sequence to specify ramp settings.

1. Select `Ramp 1` and press ON or enter the rate at which the temperature should rise. In the example in Figure 26-3, the temperature starts to rise two minutes

after the run begins. When you turn on `Ramp 1`, two more menu items appear: `Final temp 1` and `Final time 1`.

2. Select `Final temp 1`. With the numeric keypad, type the temperature that the ramp should reach and press ENTER.

3. Select `Final time 1` and type the time the oven will hold the final temperature. Press ENTER.

As soon as the `Ramp 1` parameters have been filled, a menu item for `Ramp 2` appears. If you want to program more temperature ramps, repeat Steps 1– 3 for each ramp.

NOTE

During a run, the Ramp LED will light during the first temperature rise and remain lit until the TRACE GC Ultra reaches the last ramp's final time. The Final Temp/Post Run LED will light when the last final time starts. For the example in Figure 26-3, the Final Temp/Post Run LED will light when the GC begins the 10-minute hold time for the final temperature of 250°.

Postrun Conditions

You can specify conditions for after the run. You can specify:

- an oven temperature
- how long to maintain postrun conditions
- the pressure to hold for the carrier gas

The **`OVEN`** menu settings in Figure 26-4 would be appropriate to bake out the column after a run. In the example, the GC will hold the post run temperature for 9.5 minutes, the `Post run time`, after the run is over.

NOTE

During a run, the Final Temp/Post Run LED will blink during post run conditions.

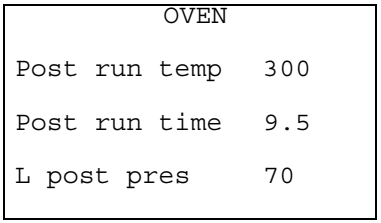

Figure 26-4. The Post Run Conditions

Other Conditions

If you want to specify other parameters, such as detector or inlet types, carrier gas pressures, autosampler parameters, or run table timed events, press the appropriate key and make changes to the menus.

For more information about detector settings, refer to Section V, *Detectors*.

For more information about injectors, refer to Section III, *Injectors*.

For more information about carrier gas, refer to Chapter 4, *Digital Gas Control*.

For more information about autosamplers, refer to Chapter 23, *AI 3000 / AS 3000 Autosampler* or Chapter 25, *Manual Functions*.

For more information about the run table, refer to Chapter 24, *Automated Functions*.

OPERATING SEQUENCE

Storing a Method

When you have specified all the conditions necessary for your analysis, press STORE, `Method`, and a number from 1 through 10. After you have completed this step, the **`METHOD`** menu will display the time and date you created the method and the number you assigned it.

NOTE If you are using a data system, you are not limited to 10 methods.

Another way to store the method is to scroll to a number in the **METHOD** menu and press ENTER.

If you were editing during a run, press EDIT/ACTIVE to return to the active mode.

27

AI 3000 / AS 3000 Autosampler Sequences

This chapter contains the instructions to programming a sample sequence with the TRACE GC Ultra keypad when an AI 3000 / AS 3000 autosampler is used and how to set up ranges of samples to run automatically.

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Operating Sequence

Running a Sequence with the AI 3000/AS 3000 496

Sequence Programming

This paragraph contains the instructions to programming a sample sequence with the TRACE GC Ultra keypad.
A *sequence* is a set of instructions for a range of samples. You can save up to five sequences in the TRACE GC Ultra. You may specify the following parameters in a sequence:

- range of samples
- analytical method to be used
- sequence repetition
- post sequence method loads

Sequence Menu Overview

To open sequence menu press SEQ. Figure 27-1 shows an example of a completed sequence menu.

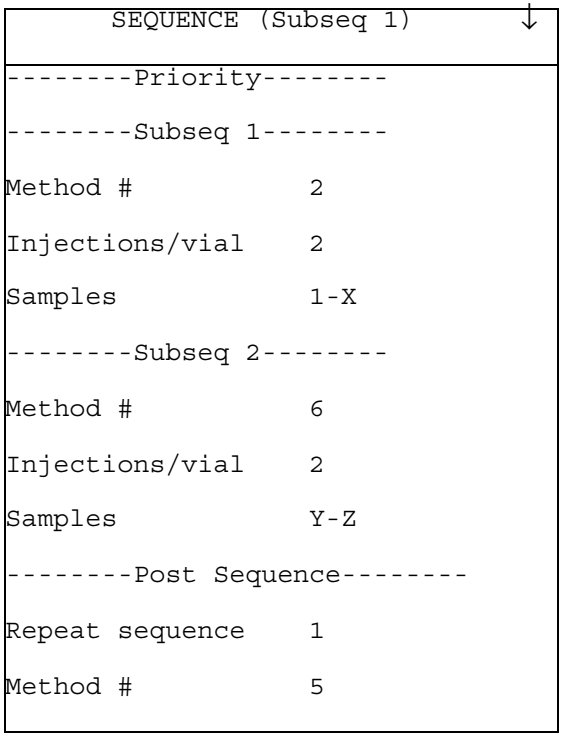
Note that each sequence has two areas of dialog:

- Subsequence, for the routine analysis of groups of samples in the tray using different methods
- Post sequence, for repeating all or part of the sequence and loading a new method

The sequence menu changes depending on your selections. After each subsequence you enter, the option for a new subsequence appears.

In the example of Figure 27-1the first subsequence directs that the first 16 samples will be analyzed by method #2 and that each sample is injected twice. The second subsequence directs that the second half of the samples is injected only once and analyzed by method #6.

After all samples have been analyzed, the post sequencing instructions call for the samples to be rerun once. When the sequence is complete, the TRACE GC Ultra will load method #5.

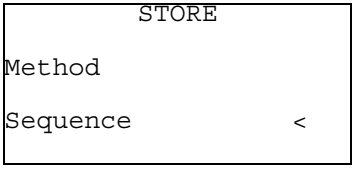


X, Y, Z Number of vials according to AI 3000 / AS 3000 sample tray

Figure 27-1. Sequence Menu

Stored Sequence Menu

To access this menu you press STORE then SEQ or STORE, then select `Sequence` and press ENTER.



The list of stored sequences, like the one in Figure 27-2, appears.

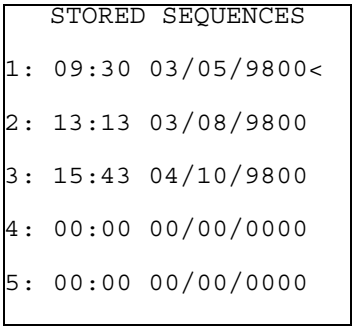


Figure 27-2. Stored Sequences Menu

This menu contains up to five rows, numbered from 1 to 5, corresponding each to a sequence.
The already stored sequences are identified by a series of numbers indicating the time and date they were created or modified. A row of zeros indicates that no sequence has been stored for that number.

CAUTION To exit **STORED SEQUENCE** menu press any key with the exclusion of CLEAR, ENTER and numerical keys.

How to Modify a Stored Sequence

NOTE You can also modify a not active sequence during a run.
Before starting, press EDIT/ACTIVE to put the TRACE GC Ultra into the editing mode.
Your edits do not affect the current run.

Select the sequence number of the stored sequence you want to modify then press any key (with the exclusion of CLEAR, ENTER and numerical keys) to exit **STORED SEQUENCE** menu.

Press SEQ, the sequence menu like the one in Figure 27-1 appears.
Modify the sequence following the instructions reported in paragraph *Sequence Set-up* then store as described in paragraph *Storing a Sequence*.

How to Create or Edit a Sequence

NOTE

You can also create or edit a sequence during a run.
Before starting, press EDIT/ACTIVE to put the TRACE GC Ultra into the editing mode.
Your edits do not affect the current run.

Select a sequence number with no data then press any key (with the exclusion of CLEAR, ENTER and numerical keys) to exit **STORED SEQUENCE** menu.
Press SEQ, the sequence menu like the one in Figure 27-3 appears.

```
SEQUENCE #1 (PRIORITY) Ø

Subseq #1          Off

Method #           1

Injections/vial    1

Samples            1-1

Subseq #2

Method #           Off

Postsequence       Off

Repeat seq         Off

Method #           Off
```

Figure 27-3. Empty Sequence Menu

Create the sequence following the instructions reported in paragraph *Sequence Set-up* then store as described in paragraph *Storing a Sequence*.

Sequence Set-up

The following sections describe how to set up each part of the sequence.

How to Set Subsequences

You can break a sequence into subsequences to specify different analytical methods and handling for various ranges of samples in the tray.

You can specify up to five subsequences in the TRACE GC Ultra.

Table 27-1. Subsequence Options in the Sequence Menu

Menu	Range	Comments
`-----Subseq 1----`		This is the title of the **SEQUENCE** menu.
`Method #`	1–9	Enter the analytical method you want to use. Refer to Refer to Chapter 26, *Using Analytical Methods*, for information about programming methods.
`Injections/vial`	1–99	Enter the number of times each sample should be run consecutively.
`Samples`	1–8 or 1–105	Enter a range of sample numbers according to AI 3000 or AS 3000 sample tray.
`Subseq 2`		Press ON to set up another subsequence.

When you have entered the required data for a subsequence, a menu for a new subsequence appears. If you do not want to add more subsequences, leave the next subsequence set to `Off`.

How to Set Post Sequence Events

When you reach the post sequence part of the menu, you have the option of repeating the sequence either a specified number of times or in an infinite loop. You can also specify that a new method be loaded.

Table 27-2. Post Sequence Section of Sequence Menu

Menu	Range	Comments
`Repeat seq  Off  <`	On/Off, 0–999, ∞	Enter ON or a range of samples to repeat the previous sequence. Choosing ON reruns the same range as specified in the sequence.
`Method #  3`	1–10	Choose a method number to load after the samples have finished all specified repetitions.

Storing a Sequence

To store a sequence after you have specified its parameters, press STORE, select `Sequence`, and enter the number you've assigned to the sequence (an integer from 1 to 5) then press ENTER.

If you were editing during a run, press EDIT/ACTIVE to return to the active mode.

Sequence Control

Use the SEQ CONTROL key for the following functions:

- to start a sequence
- to stop a sequence
- to pause a sequence
- to resume a sequence
- to check the status of a sequence

The **`SEQUENCE CONTROL`** menu change, depending on the current status of a sequence. Figure 27-4 illustrates several forms of the **`SEQUENCE CONTROL`** menu.

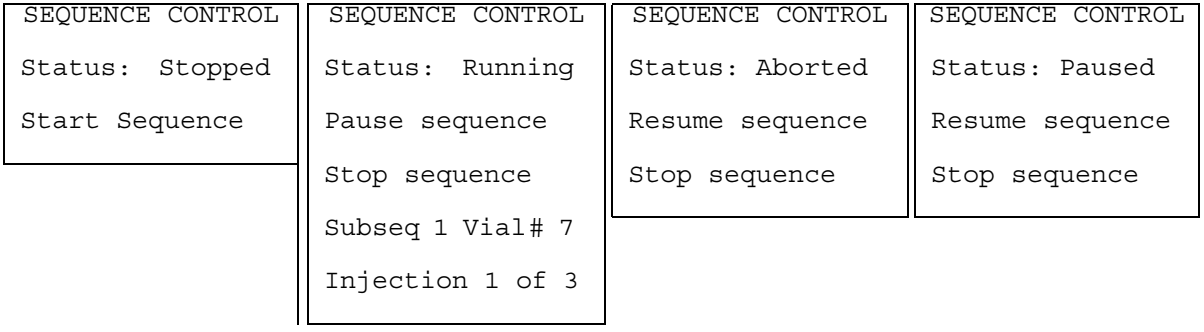


Figure 27-4. Sequence Control Menus

OPERATING SEQUENCE

Running a Sequence with the AI 3000/AS 3000

To start a sequence proceed as follows.

Loading a Stored Sequence

1. When the **Standby/Prep Run** LED is lit, press LOAD.
2. Select `Sequence` and press ENTER. The **`SEQUENCE`** menu appears.
3. Select the sequence you want to run. Press ENTER. The sequence is now loaded.

NOTE

If you have not stored a sequence, refer to *Sequence Programming* on page 489. If you press SEQ CONTROL and select `Start sequence` without having created or loaded a sequence, the TRACE GC Ultra will use the default specifications in Figure 27-3 on page 493.

Starting a Sequence

1. Press SEQ CONTROL. The **`SEQUENCE CONTROL`** menu should appear like the first example in Figure 27-4. If another sequence is running, aborted, or paused, you have the option of stopping it.
2. Select `Start Sequence` and press ENTER.

   Depending on the settings in the method you chose, the TRACE GC Ultra may return to a Not Ready state. The **Not Ready** LED will light to indicate this condition.
3. If you turned on the `Auto Prep Run` feature in the **`CONFIGURE OVEN`** menu, skip to the next step.

   If the TRACE GC Ultra is not configured to automatically do the prep run, press the PREP RUN key.

4. If you configured the TRACE GC Ultra to start when it receives a signal from an external device or programmed an automatic start in the method's run table, you need do nothing further.

   If you have not programmed the TRACE GC Ultra to start automatically, press the START key after the **Ready to Inject** LED lights.

A

Ionization Potential of Selected Molecules

Use the information in this appendix to determine the PID lamp intensity necessary to ionize certain molecules from the following groups:

- Simple Molecules
- Paraffins and Cycloparaffins
- Alkyl Halides
- Aliphatic Alcohol, Ether, Thiol, and Sulfides
- Aliphatic Aldehydes and Ketones
- Aliphatic Acids and Esters
- Aliphatic Amines and Amides
- Other Nitrogen Containing Molecules
- Heterocyclic Molecules
- Olefins, Cyclo-olefins, and Acetylenes
- Olefin Derivatives
- Aromatic Compounds
- Miscellaneous Molecules

Simple Molecules

Molecule	IP (eV)	Molecule	IP (eV)	Molecule	IP (eV)	Molecule	IP (eV)
H_2	15.46	HBr	11.62	NO	9.25	HF	15.77
N_2	15.58	HI	10.38	H_2O	12.59	H_2S	10.46
O_2	12.07	SO_2	12.34	HCN	13.91	HCl	12.74
F_2	15.7	CO	14.01	NH_3	10.15	N_2O	12.90
Cl_2	11.48	CO_2	13.79	PH_3	9.98		
Br_2	10.55	COS	11.18	PCl_3	9.91		
I_2	9.28	CS_2	10.08	AsH_3	10.03		

Paraffins and Cycloparaffins

Molecule	IP (eV)	Molecule	IP (eV)	Molecule	IP (eV)	Molecule	IP (eV)
methane	12.98	n-heptane	10.08	propane	11.07	n-hexane	10.18
ethane	11.65	2,2,4 trimethylpentane	9.86	n-butane	10.63	n-pentane	10.35
cyclopropane	10.06	cyclopentane	10.53	cyclohexane	9.88		

Alkyl Halides

Molecule	IP (eV)	Molecule	IP (eV)
methyl chloride	11.28	dichloromethane	11.35
trichloromethane	11.42	tetrachloromethane	11.47
ethyl chloride	10.98	1,2-dichloroethane	11.12
1-chloropropane	10.82	1-chlorobutane	10.67
methyl bromide	10.53	dibromomethane	10.49
tribromomethane	10.51	CH_2BrCl	10.77
$CHBr_2Cl$	10.59	ethyl bromide	10.29
1,1-dibromoethane	10.19	1-bromopropane	10.18
1-bromobutane	10.13	2-bromobutane	9.98
methyl iodide	9.54	ethyl iodide	9.33
1-iodopropane	9.26	1-iodobutane	9.21
1-iodopentane	9.19	$CFCl_3$ (Freon 11)	11.77
CF_2Cl_2 (Freon 12)	12.31	CF_3 Cl (Freon 13)	12.91
$CHClF_2$ (Freon 22)	12.45	CF_3CCl_3 (Freon 113)	11.78

Aliphatic Alcohol, Ether, Thiol, and Sulfides

Molecule	IP (eV)	Molecule	IP (eV)
methyl alcohol	10.85	ethyl alcohol	10.48
n-propyl alcohol	10.20	i-propyl alcohol	10.16
n-butyl alcohol	10.04	dimethyl ether	10.00
diethyl ether	9.53	methanethiol	9.44
ethanethiol	9.28	1-propanethiol	9.19
1-butanethiol	9.14	dimethyl sulfide	8.68
ethyl methyl sulfide	8.55	diethyl sulfide	8.43
di-n-propyl sulphide	8.30	methyl disulphide	8.46
ethyl disulphide	8.27		

Aliphatic Aldehydes and Ketones

Molecule	IP (eV)
formaldehyde	10.87
propionaldehyde	9.98
acrolein	10.10
acetone	9.69
methyl n-propyl ketone	9.39
methyl n-butyl ketone	9.34
cyclopentanone	9.26
2,3-butanedione	9.23
benzaldehyde	9.53

Molecule	IP (eV)
acetaldehyde	10.21
n-butyraldehyde	9.86
crotonaldehyde	9.73
methyl ethyl ketone	9.53
diethyl ketone	9.32
2-heptanone	9.33
cyclohexanone	9.14
2,4-pentanedione	8.87

Aliphatic Acids and Esters

Molecule	IP (eV)
formic acid	11.05
propionic acid	10.24
ethyl acetate	10.11
methyl propionate	10.15

Molecule	IP (eV)
acetic acid	10.37
n-butyric acid	10.16
n-butyl acetate	10.01
ethyl propionate	10.00

Aliphatic Amines and Amides

Molecule	IP (eV)
methyl amine	8.97
n-propyl amine	8.78
dimethyl amine	8.24
di-n-propyl amine	7.84
trimethyl amine	7.82
formamide	10.25
N,N-dimethyl formamide	9.12
tri-n-propyl amine	7.23

Molecule	IP (eV)
ethyl amine	8.86
n-butyl amine	8.71
diethyl amine	8.01
di-n butyl amine	7.69
triethyl amine	7.50
acetamide	9.77
N,N-diethyl formamide	8.89

Other Nitrogen Containing Molecules

Molecule	IP (eV)
nitromethane	11.08
1-nitropropane	10.81
propionitrile	11.84
ethyl nitrate	11.22
methyl isothiocyanate	9.25

Molecule	IP (eV)
nitroethane	10.88
acetonitrile	12.22
acrylonitrile	10.91
ethyl thiocyanate	9.89

Heterocyclic Molecules

Molecule	IP (eV)
furan	8.89
thiophene	8.86
pyridine	9.32
2,3-lutidine	8.85

Molecule	IP (eV)
tetrahydrofuran	9.54
pyrrole	8.20
2-picoline	9.02

Olefins, Cyclo-olefins, and Acetylenes

Molecule	IP (eV)
ethylene	10.51
1-butene	9.58
1-pentene	9.50
1,3-butadiene	9.07
cyclopentene	9.01
acetylene	11.41

Molecule	IP (eV)
propylene	9.73
trans-2-butene	9.13
1-hexene	9.46
1-butyne	10.18
cyclohexene	8.94
propyne	10.36

Olefin Derivatives

Molecule	IP (eV)
vinyl chloride	9.99
tetrachloroethylene	9.32
3-chloropropene	10.04
crotonaldehyde	9.73
vinyl acetate	9.19

Molecule	IP (eV)
trichloroethylene	9.45
vinyl bromide	9.80
1-bromopropene	9.30
allyl alcohol	9.67

Aromatic Compounds

Molecule	IP (eV)	Molecule	IP (eV)
benzene	9.245	toluene	8.82
ethyl benzene	8.76	n-propyl benzene	8.72
n- butyl benzene	8.69	o-xylene	8.56
m-xylene	8.56	p-xylene	8.44
styrene	8.47	naphthalene	8.12
1-methylnapthalene	7.69	anthracene	7.55
phenanthrene	8.1	fluorene	8.63
biphenyl	8.27	phenol	8.50
anisole	8.22	phenetole	8.13
benzaldehyde	9.53	acetophenone	9.27
phenyl isocyanate	8.77	benzonitrile	9.70
nitrobenzene	9.92	aniline	7.70
fluoro-benzene	9.195	chloro-benzene	9.07
bromo-benzene	8.98	iodo-benzene	8.73
benzotrifluoride	9.68		

Miscellaneous Molecules

Molecule	IP (eV)	Molecule	IP (eV)
ethylene oxide	10.56	propylene oxide	10.22
p-dioxane	9.13	acetyl chloride	11.02
acetyl bromide	10.55	phosgene	11.77
diethyl sulphite	9.68		

B

Customer Communication

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

This appendix also contains a one-page *Reader Survey*. Use this survey to give us feedback on this manual and help us improve the quality of our documentation

How To Contact Us

Use http://www.thermo.com/com/cda/resources/resource_detail/1,,12512,00.html address for products information.

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

Reader Survey

Product: TRACE GC Ultra
Manual: Operating Manual
Part No.: 317 091 70

Please help us improve the quality of our documentation by completing and returning this survey.
Circle one number for each of the statements below.

	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 install the system using this manual.	1	2	3	4	5

If you would like to make additional comments, please do. (Attach additional sheets if necessary.)

_____

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_____

Fax or mail this form to:
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Strada Rivoltana km 4
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Fax: 39 02 95059388

Glossary

This section contains an alphabetical list and descriptions of terms used in this guide and the help diskette. It also includes abbreviations, acronyms, metric prefixes, and symbols.

A

A	ampere
ac	alternating current
ADC	analog-to-digital converter

B

b	bit
B	byte (8 b)
baud rate	data transmission speed in events per second

C

ºC	Celsius
CIP	Carriage and Insurance Paid To
cm	centimeter
CPU	central processing unit (of a computer)
CSE	Customer Service Engineer

D

d	depth
DAC	digital-to-analog converter
dc	direct current
DGFC	Digital Gas Flow Controller
DCC	Digital Carrier Controller

DS	data system

E

ECD	Electron Capture Detector
EMC	electromagnetic compatibility
ESD	electrostatic discharge

F

°F	Fahrenheit
FID	Flame Ionization Detector
FOB	Free on Board
FPD	Flame Photometric Detector
ft	foot

G

g	gram
gain	A measure of the ability of an electronic circuit or device to increase the magnitude of an electronic input parameter.
GC	gas chromatograph - gas chromatography
GND	electrical ground

H

h	height
h	hour
harmonic distortion	A high-frequency disturbance that appears as distortion of the fundamental sine wave.
HOT OC	High Oven Temperature Cold On-Column 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
LVOCI	Large Volume On-Column Injector
LVSL	Large Volume Injector

M

m	meter (or milli [10^{-3}])

M	mega (10^6)
µ	micro (10^{-6})
MBq	megabecquerel
mCi	millicurie
meniscus	The curved upper surface of a column of liquid.
min	minute
mL	milliliter
mm	millimeter
MS	Mass Spectrometer/Mass Spectrometry
m/z	mass-to-charge ratio

N

n	nano (10^{-9})
negative polarity	The inverse of a detector signal polarity.
nm	nanometer
NPD	Nitrogen Phosphorous Detector

O

OCI	On-Column Injector
OD	outside diameter
Ω	ohm

P

p	pico (10^{-12})
Pa	pascal
PCB	printed circuit board

PDD	Pulsed Discharge Detector
PID	Photoionization Detector
PKD	Packed Column Injector
PN	part number
PPKD	Purged Packed Column Injector
psi	pounds per square inch
PTV	Programmable Temperature Vaporizing Injector

R

RAM	random access memory
RF	radio frequency
ROM	read-only memory
RS-232	industry standard for serial communications

S

s	second
S/SL	Split/Splitless Injector
sag	See *surge*
slow average	A gradual, long-term change in average RMS voltage level, with typical durations greater than 2 s.
SOP	Standard Operating Procedures
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.

T

TCD	Thermal Conductivity Detector
transient	A brief voltage surge of up to several thousand volts, with a duration of less than 50 µs.

U

UFM	Ultra Fast Module

V

V	volt
V ac	volts, alternating current
V dc	volts, direct current
VGA	Video Graphics Array

W

w	Width
W	Watt

NOTE The symbol for a compound unit that is a quotient (for example, degrees Celsius per minute or grams per liter) is written with a negative exponent with the denominator. For example:
$^{\circ}C\ min^{-1}$ instead of °C/min
$g\ L^{-1}$ instead of g/L

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